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14. ABSTRACT
This Test Operations Procedure (TOP) provides preparation, planning, conducting, and reporting procedures for testing sorbent-based air purification components (APCs) for the protection capability area of chemical defense. These procedures are designed to analyze the effectiveness of APCs for removing any chemical gas or vapor from incoming air. The intent of this process is to produce traceable, quantifiable, and defensible data that can be used to analyze an APC's ability to filter air in a chemically contaminated environment.

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US ARMY TEST AND EVALUATION COMMAND
TEST OPERATIONS PROCEDURE

*Test Operations Procedure 08-2-197 24 June 2016
DTIC AD No.

CHEMICAL PROTECTION TESTING OF SORBENT-BASED AIR PURIFICATION COMPONENTS (APCs)

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1. SCOPE.

1.1 Purpose.

a. This Test Operations Procedure (TOP) provides preparation, planning, conducting, and reporting procedures for testing sorbent-based air purification components (APCs) for the protection capability area of chemical defense. These procedures are designed to analyze the effectiveness of APCs for removing any chemical gas or vapor from incoming air. The intent of this process is to produce traceable, quantifiable, and defensible data that can be used to analyze an APC's ability to filter air in a chemically contaminated environment.

b. This TOP addresses testing and analysis of APCs to include chemical protective filtration materials such as media, fabrics, and canisters. APCs may be used for filtering incoming air in individual protection (IP) or collective protection (CP) systems. An IP or CP filter may be tested as a whole canister or as a mounted swatch of fabric or media. Filter fabrics (FFs) and media may have multiple layers. Fielded IP and CP filters include high-efficiency particulate air (HEPA) layers to remove dust from incoming air and to retain filtration media in the APC. The HEPA layer does not absorb vapor. The C2A1 gas mask canister is an example of an IP canister and the M48A1 and M98 particulate canisters are examples of CP canisters.

c. The procedures in this TOP cover small-scale and large-scale test fixtures. A small-scale fixture is small enough to be placed inside a laboratory fume hood. Examples of small-scale fixtures are the swatch including filter test (SWIFT) fixture[1**] and tube-filter hybrid test apparatus[2]. Large-scale fixtures are placed in a chamber to contain toxic chemical vapors. An example of a large-scale fixture is the Advanced Air Purification Test Fixture (AAPTF)[3].

1.2 Application.

This TOP provides the current standard for the planning and conduct of chemical protective performance tests of APCs. The test procedures described herein will be used as the basis for test plans. The procedures may require modification for unique items or materials or to satisfy specific testing requirements as specified in test program documentation. Procedures will be altered only after full consideration of any possible effects on the reliability and validity of the data to be obtained. Such alterations will be coordinated among all concerned organizations including the relevant Capability Area Process Action Team (CAPAT) and Test and Evaluation Capabilities and Methodologies Integrated Process Team (TECMIPT) in advance of any testing. Any alterations of procedures or deviations from this TOP will be accounted for in the test plan.

** Superscript numbers correspond to Appendix F, References.

1.3 Limitations.

a. This TOP is limited to currently approved standards, methods, and procedures. Developments in practices, equipment, and analysis may necessitate new testing procedures. Additionally, test methods and standards must be adjusted as technologies advance. Test procedures and parameters listed in this TOP may require updating to accommodate new technologies in APCs or in test instrumentation. Any updates should be described in the specific test plan.

b. The procedures in this TOP do not specify performance criteria for any specific test program. Completing a test based on these procedures does not imply acceptance or rejection of the tested APC by the U.S. Army Test and Evaluation Command (ATEC).

c. This TOP is not applicable to the testing of filters for biological or radiological contaminants, or for any contaminants in particulate or liquid aerosol form.

d. The TOP does not specify requirements, test conditions, or specific chemicals that will vary by test program. Specific values in test program requirements documentation will be used where values in this TOP conflict with the values in the test program requirements documents, so long as the test fixtures are validated to meet requirements from the Joint Requirements Office (JRO) or elsewhere.

e. Results obtained using this TOP may be compared with results from tests of other filtration technologies during the same experiment or previously measured under the same conditions, within the bounds of uncertainty (Paragraph 6.1.l). If a comparison with previous data is planned, special caution must be taken to include similar conditions in the desired comparison test.

f. The results obtained by using these test procedures under the controlled test environment conditions cannot be correlated with the full range of battlefield conditions.

2. FACILITIES AND INSTRUMENTATION.

Facilities and instrumentation requirements specified in the following paragraphs will be met. The facilities and instrumentation selected must be identified and described in the test plan.

2.1 Facilities.

Chemical laboratory and chamber facilities are required for APC testing and must conform to all federal and state regulations and relevant required policies for handling and storing the chemical materials of interest. Testing facilities intending to use chemical warfare agents (CWAs) and nontraditional agents must adhere to the terms of the Chemical Weapons Convention (CWC)[4] for storage and handling of agents[5,6,7]. The toxic chemical laboratory and chemical agent storage facility selected for use in testing must provide secure storage, handling, analysis, and decontamination capabilities.

Item	Requirement
Chemical agent storage facility	The storage facility must be constructed to ensure safe and secure storage, handling, and decontamination capabilities for research, development, test, and evaluation (RDT&E) quantities of chemical agents.
Chemical agent laboratory	Testing with agent must be conducted in an agent-certified facility. Examples of agent-certified laboratories are the Reginald Kendall Combined Chemical Test Facility (CCTF) at U.S. Army Dugway Proving Ground (DPG), Utah, and the Containment Facility at Edgewood Chemical and Biological Center (ECBC), Aberdeen, Maryland. The chemical agent laboratory and personnel assignments must meet all requirements of Army Regulation (AR) 50-6[5], AR 190-59[6], and the safety requirements of U.S. Army Materiel Command Regulation (AMCR) 385-100[7] and Department of the Army (DA) Pamphlet (PAM) 385-61[8].
Environmental chamber	Testing chambers must be located within chemical agent laboratories, integrated with adequate test fixtures, and advanced engineering systems to control key environmental variables, including temperature, relative humidity (RH), airflow, and air pressure, to meet the test requirements while protecting operators and the external environment from exposure hazards. An example of an environmental chamber suitable for APC testing is the Toxic Test Chamber (Environmental Chamber) at ECBC.
Medical clinic	The testing facility must have the medical facilities, authorities, and equipment required to treat exposure to chemical agent, overexposure to simulant, or adverse reactions to physiological stress. Emergency medical technicians (EMTs) will be available during all chemical agent trials to respond to possible adverse physiological responses in participants and to provide appropriate medical aid whenever necessary.
Battlefield contaminant (BFC) exposure chamber (if required)	A BFC exposure chamber is an environmental chamber or other suitable structure into which BFC vapors and aerosols can be disseminated to expose APCs before chemical testing. BFC exposure chambers must have relief openings to prevent backpressure. Effluent from the APCs must be vented out of the BFC exposure chamber to prevent the recirculation of BFC components that are poorly filtered by the APC. Recirculation may also increase the humidity of the BFC exposure chamber. For some BFCs, scrubbers or filters (in addition to the APC) may be required to contain escaping BFC components.

2.2 Instrumentation.

a. Detectors are required to monitor challenge, effluent, and concentrations of the chemicals used during APC testing. Other instruments are required to monitor the laboratory or chamber environmental conditions. It should be determined whether toxic byproducts are produced. If necessary, the effluent should be monitored for toxic byproducts as well as for the challenge compound.

b. Real-time monitors (RTMs) widely used to monitor chemical vapor or gas concentrations include ion mobility spectrometers (IMSs) or Fourier-transform infrared (FTIR) gas analyzers. Gas chromatographs (GCs) with integrated pre-concentrators are near real-time monitors (NRTMs) widely used to monitor chemical vapor or gas. An NRTM is an RTM with a sample interval of 15 minutes or less, preferably one minute or less. Manufacturer-provided operating instruction procedures and pertinent ARs must be followed for instrument calibration, calibration check, routine inspection, and maintenance, according to equipment maintenance methods and must ensure that calibration meets the criteria for precision and accuracy indicated in the test organization's technical specification. Instrument calibration will be traceable to standards maintained by the National Institute of Standards and Technology (NIST). Instruments should be operated in accordance with (IAW) the instrument manufacturer's recommended operating parameters or approved laboratory standing operating procedures (SOPs). Any measuring device meeting the needs in Paragraph 2.2 may be used. This TOP does not mandate the use of a specific technology. The use of brand names does not constitute endorsement by the Army or any other agency of the Federal Government, nor does it imply that it is best suited for its intended application.

Parameter(s)	Measuring Device	Permissible Error of Measurement
Challenge concentration	RTMs or NRTMs for a detection range from approximately 1 to 5,000 mg/actual m^3. For compounds that are difficult to detect and quantify it is advisable to use two different technologies in parallel. The FTIR gas analyzer is suitable for infrared-active compounds[2]. Other potential and commonly used instruments may include GCs equipped with flame ionization detector (FID), flame photometric detector (FPD), or thermal conductivity detector (TCD). A low-volume sampling (LVS) loop may be used together with a GC. It may be necessary to dilute the challenge by a known	±15 percent at all test conditions and in the presence of other compounds on the test item as shown by error bars on results.

Parameter(s)	Measuring Device	Permissible Error of Measurement
Challenge concentration (Cont'd)	ratio for accurate measurement for some chemicals. Mass balance should only be used as a last resort in verifying challenge concentration; it should be noted that a mass balance can be misleading because of surface loss, misting, pooling, etc.	
Effluent concentration	RTMs or NRTMs. The required concentration range depends on test requirements. Methods used for these monitoring systems must be validated before use. Agents and simulants may be monitored by a GC equipped with a FPD and/or FID interfaced with a pre-concentrator. Other potential and commonly used instruments may include GCs equipped with TCD and MINICAMS®. Different technologies may be used[2]. Toxic industrial chemicals (TICs) such as cyanogen chloride and hydrogen cyanide can be detected using a GC equipped with a FID or nitrogen-phosphorus detector (NPD). Arsine may be detected using a GC equipped with a TCD, FTIR, or hydride detector.	±20 percent at all test conditions and in the presence of other compounds on the test item.
Data acquisition/ processing	Data acquisition system (DAS). For example, hardware and software from National Instruments (Austin, Texas), and Opto 22 (Temecula, California), were used to produce accredited results[1]. The selected DAS should be managed IAW hardware and software configuration management.	The DAS computer system should record data from all instruments that have either a digital or analog output. Values should be displayed to the operator. **NOTE**: Considering sources of error from the electronic operation of the DAS, the error is estimated as ±0.1%.

Parameter(s)	Measuring Device	Permissible Error of Measurement
Humidity	Humidity probe. The Vaisala HUMICAP® models 333 and 337 probes (Vaisala, Boulder, Colorado) as well as probes from Edge-Tech Instruments (Marlborough, Massachusetts) were used to produce accredited results[1,2].	±2 percent RH or the corresponding range for water vapor content (WVC). **NOTE**: RH is more widely used and instruments read in RH. WVC does not vary with temperature and better conveys the water content of the air.
Challenge flow rate and flow rate through each test item (if applicable)	Mass flow meters or hot wire anemometers	±5 percent
Barometric pressure	Barometer	±1 mbar
Challenge gas inlet temperature and test cell temperature (for small-scale testing as described in Paragraph 4.5.2)	Thermocouple or resistance temperature detector (RTD)	±1°C
Differential pressure (ΔP) across the test item	Pressure transducer	±0.01 inches water gauge (iwg)
Filter mass before and after testing	Scale	±0.01 percent

2.3 Test Controls.

Unless otherwise specified, the following tolerances will be met. **NOTE**: These are the testing tolerances, which are larger than the error in measurement (Paragraph 2.2).

Parameter	Tolerance
Challenge inlet temperature	Maintain the target ±3°C.

Parameter	Tolerance
Challenge inlet humidity	Maintain the target ±5 percent RH or the corresponding range of WVC.
Challenge concentration	Maintain the target ±15 percent.
Background challenge concentration	Before a trial, the background challenge concentration must be <1 percent of the target challenge concentration.
Background effluent concentration	Before a trial, the effluent concentration must be less than 10 percent of the breakthrough concentration.
Challenge flow rate	Maintain the target ±5 percent.
across the test item	Maintain the target ±0.02 iwg if ΔP control is required.

3. REQUIRED TEST CONDITIONS.

3.1 Documentation.

3.1.1 Familiarization.

a. Potential problem areas must be identified by reviewing records and results of similar tests, if available.

b. Development of test plans requires familiarization with the applicable test planning and requirements documents such as:

(1) Safety release and approval from the authorizing agency (e.g., ATEC) to begin testing, if required.

(2) Human use committee (HUC) approval or exemption and notification, if required.

(3) Government and manufacturer's publications, including the current safety data sheets (SDSs) for all materials used in the test.

(4) Program-specific requirements documents: capability development document (CDD), system performance specification (SPS), system evaluation plan (SEP), safety assessment report (SAR), test support order, event design plan (EDP), system support package (SSP), and SSP list (SSPL).

(5) Industrial hygiene plan (IHP).

(6) Familiarization with test reports of similar and related programs to avoid unnecessary duplication of effort.

c. Test personnel must familiarize themselves with the relevant SOPs and other procedures for applicability, completeness, and adequacy. These documents will be updated as required.

d. All applicable/available safety documents such as the SAR and health hazard assessments (HHAs) should be reviewed to determine if any safety or health issues require special test protocols.

e. Test personnel must familiarize themselves with vendor-provided user manual(s) for the APC.

3.1.2 Environmental Compliance.

a. In compliance with the National Environmental Policy Act (NEPA), the Department of Defense (DOD) requires that an environmental impact assessment for the life cycle be prepared and that potential environmental impacts be assessed at the earliest possible stage in the planning process.

(1) When the proposed test may significantly affect the environment, is environmentally controversial, or when litigation is expected based on environmental issues, a detailed environmental impact statement will be prepared by the test center and analyzed IAW the NEPA processes.

(2) When a review indicates that there is existing NEPA documentation already in place for the proposed test, a record of environmental consideration (REC) will be completed for the test. The REC will indicate the process for consideration of environmental concerns and rationale for the conclusion.

b. Test personnel and participants must receive and understand environmental documentation before the test begins.

3.2 Test Planning.

a. The test plan will be written before test execution and will incorporate input from test and evaluation subject matter experts together with material subject matter experts. Any amendments must be documented and approved by the test center management in concurrence with the customer. Test plan format can be test-site specific with customer agreement but the

content will address all elements required for test conduct. The following elements must be considered.

(1) The test plan must refer to the data management plan (DMP) that describes data collection and reduction analytical procedures and reporting procedures.

(2) The test plan must include safety procedures addressing hazard analysis, operations, and decontamination. Safety procedures should provide for a test readiness review (TRR) and test center operational readiness inspection (ORI) before testing begins.

(3) The test plan must define the required challenge and effluent concentration ranges of the test chemical, accounting for the trial conditions and the chemical's physicochemical properties, analytical limitations, and safety considerations.

(4) The test plan must identify suitable chemical challenge systems and referee instrumentation based on the chemical of interest, concentration, and environmental conditions.

(5) The test plan may include plans for contingencies and deviations, but implications of any such deviations must be analyzed, documented, and presented in the final test report.

(6) The test plan may include intended report recipients if known, so that final report writers will know the intended audience.

b. Simulant Selection.

(1) Simulants are often employed in lieu of agents during testing and evaluation of chemical and biological (CB) systems to mitigate the risks associated with the use of agents. Simulants may have chemical or physical properties that closely mimic those of agents. Simulants may have an ability to mimic the chemical or physical mechanisms of interest for agents in a given environment. Simulants may be less toxic, less expensive, and have less environmental impact than agents. In addition, simulants do not have surety restrictions. No simulant will completely match the agent in all respects.

(2) Simulants should be verified and validated before use in APC testing. Selection of simulants should be conducted IAW TOP 08-2-196[9]. The objective will be framed, potential simulants will be identified and screened, simulants that best mimic the desired agents will be selected, and the selected simulants will be verified and validated. For APC testing, selected simulants need to mimic the adsorptive or reactive behavior of an agent. APC tests may require the use of a novel simulant to mimic particular characteristics of the agents of interest.

c. Experimental Design. A design of experiment (DoE) is strongly encouraged for developing test methods, agent-to simulant relationships, and empirical models because statistical validity of such data is required[10] (Paragraph 4.4.1). Properly planned trials minimize the number of trials needed to obtain statistical validity.

3.3 Safety.

3.3.1 General.

a. All test operators must read, understand, and have available the SDS associated with each chemical used in the test and with each material in the filter bed. The operator is expected to be familiar with the operation of the test fixture and to have read and understood the test plan. The test plan will be available to the operators at the test site.

b. The required SDSs, testing protocols, and safety procedures will be available at the test site.

c. When appropriate, the test personnel will wear required personal protective equipment (PPE). PPE must be approved by the Office of the Director of the Army Safety (ODASAF, Fort Belvoir, Virginia) or meet certification standards from Occupational Safety and Health Administration (OSHA, Washington D.C.) and National Institute for Occupational Safety and Health (NIOSH, Atlanta, Georgia) Chemical, Biological, Radiological, Nuclear (CBRN) certification standards.

d. Medical examinations of test participants may be required to determine physical ability to perform specified tasks. Medical examinations will be conducted before the test begins. If applicable, a medical record will be maintained on each participant.

e. Test personnel will be informed of potential safety and health hazards involved in test conduct and the precautions required to prevent accidents and over-the-limit exposure to the simulant used in the test. A safety survey will be conducted before test execution. Other health risks may include slips, trips, falls, and exposure to hot or cold surfaces or liquids.

f. Test personnel must submit to a physical examination and must be certified by onsite medical personnel for eligibility to perform test assignments.

g. Daily safety checks and briefings will be conducted to ensure that all identified safety hazards have been addressed before testing proceeds.

h. For tests that involve carrying or lifting, test personnel and participants will be instructed in the proper lifting procedures.

i. Fixtures will be controlled remotely wherever applicable. A barrier will separate the operator from the fixture to protect operators from exposure to fixture hazards.

3.3.2 Chemical Toxicity.

a. This procedure may be used with toxic chemicals such as agents and TICs. Even simulants, which are less toxic than agents, may be disseminated during testing at concentrations that may be hazardous to personnel. All handling of toxic chemicals should be performed within well-ventilated areas. The operator must wear PPE, including but not limited to ocular, dermal, and respiratory protection IAW applicable SOPs.

b. The analysis of chemical toxicity issues must include consideration of the effects of leaks or spills on both the test operator and nearby laboratories. Particularly for large-scale tests, large quantities of chemicals may be required and the effect of leaks and spills may be substantial. Storage and handling SOPs must be developed (if not already in place) and followed.

c. Personnel trained in the handling of agents will handle agents with extreme care at an approved surety installation. Surety regulations must be followed.

d. The discharged vapor concentration should be reduced to the level required by the institution safety requirements.

e. Safety air monitoring systems and hazardous operation procedures for handling agent must be established IAW the safety and security requirements from each applicable DA regulation in order to execute the test in an efficient, effective, and safe manner[7,8,11].

3.3.3 Fire, Pressure, and Explosion Hazard.

a. Many filters contain activated carbon impregnated with copper, silver, zinc, molybdenum, and triethylenediamine (ASZM-TEDA), which is flammable. Handling and disposal of filtration media may release explosive dust. Test personnel must check the SDSs of all chemicals for flammability and explosive hazards. Reactive chemicals, such as arsine, phosphine, nitrogen dioxide, phosgene, chlorine, hydrogen chloride, and hydrogen cyanide, have been known to ignite filter material. A fast exothermic reaction is likely when high challenge concentrations are used. It is best to consult an expert when conducting a fire risk assessment. The filter used in testing is not the only filter that may be at risk; sacrificial and building filters may be made from similar materials.

b. If an unexpected temperature increase occurs in any filter, the test should be immediately stopped and the cause of the change determined. In particular, if the effluent temperature is more than 30°C higher than the inlet temperature, immediate action is critical. Ignoring an unexpected temperature change could cause a fire, an explosion, or an equipment failure that may result in injury or death.

c. Fire extinguishers of some types may be prohibited by the SDS of the test APC. Filter fires should be extinguished IAW test center SOPs. For example, carbon dioxide fire extinguishers are not effective on burning carbon filters, which may be quenched by a flow of nitrogen. After fire suppression, the filter should be dunked in water to cool hot spots. The water should be analyzed for test chemicals and other hazardous compounds and then disposed of as hazardous waste. An automatic fire-suppression system appropriate for the materials present during test could be installed.

d. Depending on the chemical type, concentration, and airflow rate, vaporization of liquid chemicals may pose a flammability or explosion hazard. If the chemical is flammable, vaporization must be performed with care to ensure that a fire does not occur. Dissemination systems must not be left in a pressurized state when not in use.

e. Heaters and pumps should be interlocked to contain chemicals in case of a loss of carrier gas pressure, electricity, duct flow, engineering controls, cooling water, computer control, or any other loss or abnormality that might create a hazard. Overpressure should be relieved into a capture device. Check valves should be used to prevent undesirable chemical backflow. The equipment should be purged thoroughly before shutting it down so that minimal chemical remains.

3.3.4 Training.

Test personnel must be trained regarding the test items, test scenarios, and test conditions to include the following:

a. Demonstration of the test item operation, training for operation of the test item, and discussion of any special characteristics and differences from comparable APCs.

b. Identification of appropriate test personnel and processes to report any safety, surety, security, or health-related issues.

3.4 Quality Assurance (QA) and Quality Control (QC).

3.4.1 General.

a. Chamber and laboratory instrument maintenance and periodic calibration with careful documentation of calibration procedures ensure the quality of test data. Operations will be conducted IAW the approved protocols of the test facility.

b. Calibration procedures should meet the guidelines of American National Standards Institute National Conference of Standards Laboratories (ANSI NCSL) Z540-3[12] or International Organization for Standardization (ISO) 10012:2003[13]. Flow resistance instruments [orifices, venturis, flow nozzles, and American Society of Mechanical Engineers (ASME) flow meters] can all be precalibrated IAW ISO 5167-1:2003[14] and ASME guidelines[15]. In the absence of a validated protocol, calibration will be conducted as directed by the instrument manufacturer or AR Technical Bulletin (TB) 750-25[16], U.S. Marine Corps Technical Instruction (TI) 4733-OD/1[17], or U.S. Air Force Technical Order (TO) 00-20-14[18].

c. Test plan, data collection sheets, technical manuals (TMs), operating manuals (OMs), training, preparation and storage procedures, labeling, and chain of custody of samples and reagents must be reviewed to ensure that all documentation requirements are fulfilled. Because QA/QC practices differ among laboratories, this TOP outlines the QA/QC elements that should be addressed by test site quality system reviewers, rather than prescribing the actual practices. QA/QC practices may be prescribed based on the need for compliance with specific standards (e.g., Good Laboratory Practice). Certain testing laboratories may also be required to comply with ISO/International Electrotechnical Commission (IEC) 17025[19].

d. If the test chemical is used as-is, then the supplier-provided certificate of purity analysis must be used as referee data.

e. Standard solutions must be prepared with an approved method that has been validated using a precision and accuracy study (at DPG this method is described in SOP DP-0000-M-073[20]).

f. Validation will be performed before testing, by conducting pilot trial(s) using APCs similar to those tested, to ensure that all test instrumentation and data collection systems are operating properly. The pilot trial results must match those previously established[1,2,3], within the limits of uncertainty.

g. Instruments will be checked before each trial with appropriate standards to ensure they are operating within statistical control [Paragraph 3.4.3.a(2)]. It is important that the control limits of RTMs are small enough to achieve the required tolerance. The zero level will be checked daily to ensure that it has not been altered by electronic drift.

h. Data will be independently reviewed, verified, and validated.

i. The serial number of each instrument, method names, start and stop times, the codes used to identify trials, the codes used within the instrument software to identify the chemical, and the calibration range of the analyzer will be recorded. If a multiport sampler is used to sequentially sample the effluent flow from different APCs, data should be recorded to associate the effluent data with the port on the sampler, and thus with the test item. If data are collected from sources that do not have outputs for connection to the DAS, they must be recorded on a hand-recorded data sheet during the test.

3.4.2 QA System.

a. A QA system will be implemented to manage routine internal and external audits and inspections and to perform corrective and preventive measures. Quality management will ensure all records are legible and available for inspection. Data packages must contain sufficient information to identify factors affecting uncertainty and enable the test to be repeated under conditions as close as possible to the original test[21].

b. Each test facility's QA program must ensure that data of the required quality are obtained from each test. The data quality objectives (DQOs) must be established by the customer and test facility's QA/QC test procedures and manuals. Measurement quality indicators (MQIs) are data properties used to analyze the DQOs. At a minimum, MQIs must demonstrate that measured data meet the DQOs established in the permissible errors of measurement described in Paragraphs 2.2 and 2.3. The procedure to determine if the DQOs are met is in Paragraph 3.4.3.

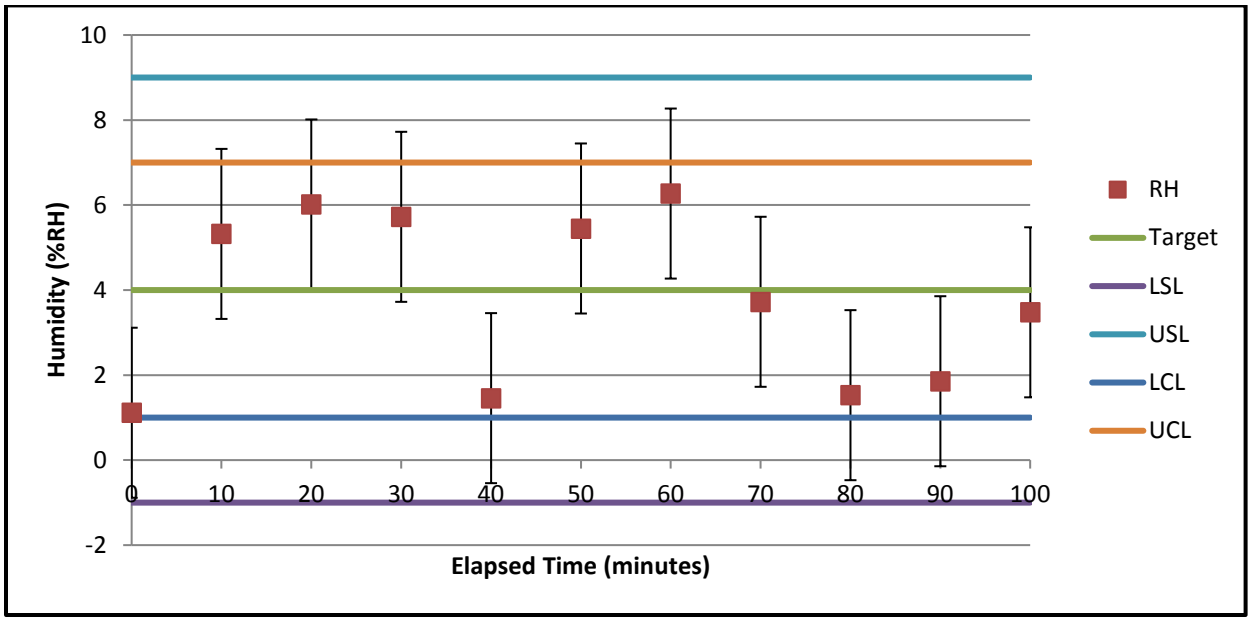
c. Safeguards will be maintained to prevent data loss and corruption. Electronic data should be backed up weekly onto a network storage system or onto removable media stored outside the laboratory. Paper laboratory notebooks may be copied or scanned to portable document format (PDF). Scanned PDF copies of laboratory notebooks should be stored on a network storage system or removable media stored outside of the laboratory. All records will be stored under proper classification for security and to ensure customer confidentiality and to prevent unauthorized access.

3.4.3 QC System.

a. DQOs.

(1) Test condition requirements indicating range, accuracy, and precision required for control and measurements should be specified in the DQOs within the test plan or standard methods. When developing a DQO, test program criteria should be reviewed together with the instrument calibration, specification data, test methods and previous fixture verification and validation (V&V) documentation, if available.

(2) DQOs should be defined before testing so that the program may be designed to provide sufficient data quality. DQOs must address quantitative and qualitative criteria that clarify study objectives and define data to collect, and specify tolerance thresholds. The resulting data must be adequate to analyze the test item (Paragraph 3.3.4). For example, dissemination DQO may be expressed as X±Y mg/actual m^3 (am^3), which represents the upper specification limit (USL) and the lower specification limit (LSL) (see Figure 1).



NOTE: RH – relative humidity; LSL – lower specification limit; USL – upper specification limit; LCL – lower control limit (Equation 1); UCL – upper control limit (Equation 2).

Figure 1. Example specification and control limits based on the measurement error and tolerance.

(a) Units of Flow and Concentration Measurements. Actual, standard, and normal units are widely used. Each test should use one consistent set of units for flow and concentration, and specify the standard temperature used, if any.

NOTE: Depending upon the instrument configuration, care must be taken to annotate the appropriate units of flow and concentration (e.g., FTIR analyzers report actual concentration whereas MINICAMS® units report standard concentration). Test documents must state explicitly which units are used to measure flow and concentration. The amount of gas may be expressed in actual units, the volume actually occupied at the temperature and pressure of the trial. The volume that the gas occupies at standard temperature and pressure may be expressed in standard units. Because equal masses of air occupy the same volume at standard conditions, a standard volume is effectively a mass. Standard units may be prefixed with the letter s, such as standard liters per minute (sLpm). Standard pressure is 1 atm. Different standard temperatures are in use. Some laboratories use 0°C, NIST uses 20°C, others calibrate instruments at 21°C, and others refer to 25°C. If the standard temperature is 25°C, the units are called normal, such as normal liters per minute (NLpm). Units of actual volume may be prefixed with the letter a, such as aLpm. Normal and standard volume may be converted to actual volume using Equation 1.

$$V_a = V_s \times \left(\frac{T + 273.15}{T_s + 273.15}\right) \times \frac{1013.25}{P} \qquad \text{Equation 1}$$

Where,

V_a = the volume in actual liters (aL)
V_s = the standard or normal volume in sL
T = the mean trial temperature in °C
T_s = the standard or normal temperature in °C
P = the mean trial barometric pressure in mbar

(b) Fixture control limits are derived from the permissible error and the tolerance established during fixture V&V (Paragraph 7). If the tolerance is less than the error of measurement, meeting the required tolerance will not be possible. If the tolerance is greater than the error of measurement, control limits can be set using Equations 2 and 3.

$$\mathrm{UCL} = V + |c| - |e| \qquad \text{Equation 2}$$

$$\mathrm{LCL} = V - |c| + |e| \qquad \text{Equation 3}$$

Where,

LCL = lower control limit
UCL = upper control limit
c = required tolerance allowed for each measurement (Paragraph 2.3)
V = target value for the test parameter from test plans
e = error of the instrumentation measuring the test parameter (Paragraph 2.2)

NOTE: If values are available from V&V, the V&V values should be used instead of the values in Paragraphs 2.2 and 2.3. If the LCL is less than zero and this is not physically possible, then zero should be used.

(c) The variability of parameters during a trial can be analyzed by plotting the value against the test time on control charts where average and the variability limits are depicted as horizontal lines (Figure 1). Control charts or other similar techniques may be used to ensure data quality.

(3) The DQOs must be defined in the test plan and should drive the selection of test methods and instrumentation. If any DQOs are not met, the trial must be repeated until the DQOs are met or the test manager grants permission to not execute the trial. To be considered valid, a trial must meet the following criteria:

(a) All required data must be collected.

(b) Challenge data (temperature, humidity, concentration, and flow rate) must be within the control limits throughout the trial. Control limits must be set to ensure that all parameters are controlled within the requirements specified for the test.

(c) The trial must be executed for the required time duration.

(d) The challenge NRTM output will be monitored to ensure that the test chemical is present in much greater amounts than any impurities, degradation products, or stabilizers. This also serves as a qualitative check of the stated agent purity and proper dissemination.

(e) The effluent NRTM output will be monitored to ensure that no BFCs or interferents impair quantitation of the test chemical.

(f) Background levels must be less than 10 percent of the breakthrough criterion concentration before the start of trials for agents and TICs. The breakthrough concentration may be based upon toxicological data such as the 10-minute upper marginal military exposure

guideline (MEG) level[22]. For simulant, the breakthrough criterion for the corresponding agent should be used.

b. MQIs.

(1) MQIs ensure accurate data collection, transcription, and manipulation. MQIs associated with data reporting are sample collection documentation, tracking, evaluation of analytical results, and comparison of results. MQIs must be detailed in the test plan and follow the test facility's QC plan. Any problems associated with a particular sample will be noted on the appropriate log sheet or data file and evaluated. All data collected will be time stamped.

(2) The technical quality of the data must be reviewed to verify the accuracy and to ensure the collected data meet the customer's requirements as defined in the test plan and other associated plans.

(3) For each trial, the vapor concentration, temperature, and humidity at all sampling locations will be measured, recorded, plotted on a chart, and compared with the tolerances in Paragraph 2.3.

(4) Statistical analysis will be used to summarize and ensure the quality of the trial data.

3.5 Provisioning.

a. Test preparations include selection and training of qualified test personnel if applicable.

b. Analytical test methodology should be developed and validated before test execution is scheduled to begin (validation is described in Paragraph 7).

c. All test participants must read and indicate that they understand the risk assessments, test plan, and test-specific procedures. The required SDSs, testing protocols, and safety procedures must be available at the test site.

d. Challenge chemicals including any agents, TICs, and simulants will be acquired with certificates of analysis (CoAs). CoAs will specify chemical purity. Test chemicals must be stored IAW laboratory procedures.

e. Agent standard solutions must be traceable to chemical agent standard analytical reference material (CASARM) standards as available. At the time of issuance, agent standard solutions must be accompanied by the most recent purity analysis results. Information will include apparent agent purity and estimated impurity amounts based on the analysis. CASARM standards are to be used by their expiration dates as specified on the CoA.

f. Whenever possible, certified reference materials (CRMs) for TICs and simulants must be NIST-traceable and stored IAW laboratory procedures. CRMs can be solutions or gas mixtures containing a known concentration. CRMs can also be solid adsorbent containing a

known amount of TIC or simulant. These CRMs must be accompanied by a CoA and must be used by the expiration date.

4. TEST PROCEDURES.

4.1 General.

a. The following paragraphs provide minimum requirements for testing procedures. Methods that are not documented in this TOP will be detailed in the test plan.

b. Test Site Readiness and Approval. A preoperational safety survey (POSS) and ORI will be arranged at the discretion of test facility management. The survey and inspection will cover all operations that will be performed during the execution of the test project. The test team must present all test-related documentation including test plan(s), risk assessments, and SOPs. Test site authorization is required before the team can proceed to test execution.

c. Test Execution. Trials must be conducted IAW the test plan.

d. Data Processing. Data analysis will be performed IAW the DMP and verified and validated by data authentication group or equivalent designated independent reviewers.

e. Reporting. The test report will be prepared, reviewed, and approved for release.

f. Retrograde. The test location will be restored and decontaminated IAW the test plan and all test documentation will be archived. All recoverable chemical test materials will be accounted for and returned to storage locations.

4.2 Receipt Inspection.

Each testing APC/material delivered will be inspected for integrity before the onset of testing. Inspection should be carried out in a toxic-free and contaminant-free environment. Ambient temperature and humidity should be recorded.

a. Visual Inspection. Visual inspection will be conducted to verify that the APC/material provided is intact and shows no evidence of tampering. The APC packaging will also be examined for any indications of damage. Any damage will be recorded, and photographs can be taken to further document the damage. Unit serial numbers and any time-stamped data such as packing date will also be recorded.

b. Identification and Coding. Each APC under test should be assigned a unique test item control number (TICN). The TICN can be generated during test preparation as sequential alphanumeric codes that identify the specific test item. Alternatively, the manufacturer's National Stock Number (NSN), product number, batch number, serial number, or lot number may be used as the TICN. The TICN must be permanently marked or attached to the test item and will be used for tracking from initial receipt through all testing.

c. Storage. Test APCs should be kept in sealed containers until needed for preconditioning (if applicable) or testing. When removed from storage, any damage to the packaging or APC will be recorded and photographed. Damage may include dents, holes, rips, tears, evidence of tampering, and loose filter media.

4.3 In-House Solution Preparation.

Calibration and QC solutions will be prepared in-house by stepwise dilution from standard solutions. Calibration and QC solutions may be prepared from the test chemical when NIST-traceable or CASARM standard solutions are not available. However, the solution concentration must be determined quantitatively using the site-specific approved analytical methods.

4.4 Test Preparation Procedures.

4.4.1 Design of Experiment.

a. Test experimental design must consider the limits of chemical vapor delivery under humid conditions. Hydrolysis of agent (reaction with water) may cause the actual agent concentration in the chamber to be less than the calculated concentration. Empirical data can be used to estimate the hydrolysis during the test. Losses from hydrolysis will be less than 10 percent under most conditions tested for nerve (G-) and blister (H-) series agents, but for persistent nerve agent (VX) and Lewisite (L) hydrolysis losses will be greater than 10 percent.

b. Test experimental design should include target values for temperature, challenge concentration, , humidity, BFC exposure (if required), and prior APC wear. Conditions should cover the range of combat developers' requirements, while lying within the range over which fixtures have been validated. One temperature should be between 20° and 30°C to test the APC at the most common operational temperature. At least one other temperature should be tested, differing at least 10°C from the first temperature. Target challenge concentration may vary from approximately 0.001 to 100,000 mg/am³, depending on compound and conditions. For a liquid chemical, the challenge vapor concentration must be less than 10 percent of the saturated vapor concentration. The risk is that a near-saturated vapor of challenge chemical will condense; thus the APC is challenged with a mist instead of a vapor. Temperature may vary with location by a few degrees because of adiabatic expansion. If the concentration is <10 percent of saturated, the vapor will not condense. Flow rates may vary across a wide range.
F FF

NOTE: The relationship of airflow through each APC to ΔP is expected to be linear[23]. The ratio of ΔP to airflow is known as the impedance and is characteristic of the APC. The impedance may change slightly during the trial if the APC is clogged. Either ΔP or airflow rate may be used as a trial target.

c. If the media are designed to emulate a specific filter, before starting the test, the required flow rate must be calculated. The flow rate through the APC is scaled to produce the same residence time as the emulated filter, where the residence time equals the bed depth divided by the air velocity or bed volume divided by flow rate. The required flow rate for a media test can be calculated in Equation 4, if not provided by the customer. To scale flow rate for two

APCs with similar media, it is assumed that media in both APCs has approximately the same void fraction. Computational modeling of airflow through the filter may suggest other approaches.

$$Q = A\frac{Q_{filter}}{A_{filter}} \qquad \text{Equation 4}$$

Where:

Q = required flow rate for media test in actual liters per minute (aLpm),
A = volume of media bed in cm^3
Q_{filter} = required flow rate through APC in actual liters per minute (aLpm) based on the operationally realistic breathing needs of the warfighters using the APC
A_{filter} = bed volume of filter media bed in cm^3

4.4.2 Test Fixture and Instrument Setup.

a. Figure A.1 shows the functional components of an APC test fixture. The required temperature will be established and stabilized by adjusting set points. The final set points and actual temperature will be recorded. Real-time temperature data should fall within the DQOs. Depending on the test, APCs may also be equilibrated to the test humidity before testing. Equilibration may be conducted on a laboratory bench, in a separate fixture, or in the test fixture.

b. Humidity probes will be used to measure the humidity in the challenge air stream. When required, humidifiers will be interfaced with controlling mechanisms to maintain humidity at the level specified within the DQOs. Humidifiers will be filled with deionized (DI) water. The mass of water in the humidifier at the beginning of the test may be recorded to allow later estimation of humidity from the amount of water remaining in the humidifier. Humidity can be adjusted by changing the temperature, flow rate of the air entering the humidifier, or flow rate of dilution air.

c. Airflow through the APC across the APC will be measured to confirm that it is within the DQO or APC design specifications. The carrier gas must be clean dry air or nitrogen. The carrier gas stream must be controlled by calibrated mass flow controllers (MFCs) and measured using flow meters equipped with appropriate valves. Large airflow rate measurements may be made by means of hot-wire anemometers.

d. Ambient barometric pressure should be recorded.

e. Gauge pressure inside the fixture should be recorded for a closed system.

4.4.3 Challenge Generation Setup.

Challenges may be generated as vapor from liquid chemicals or as diluted chemical gases.

a. Chemical Vapor Generation from Liquid Chemicals.

(1) Syringe pump setups and sparger or saturator cells may be used to generate vapor challenges from liquid chemicals. Other verified and validated dissemination systems may also be used.

(2) In a syringe pump setup, the pump will be filled with sufficient test chemical for the expected duration of the trial and the mass of the chemical fill will be recorded. The pump will be set to the desired infusion rate, which is calculated before dissemination. The infusion rate of the pump should be adjusted to maintain the concentration specified within the DQOs. The chemical will be dispensed by the syringe pump onto a heated block or tee from which it will be vaporized.

b. Generation of Vapor from Sparger or Saturator Cell.

(1) The sparger or saturator cell will be filled with sufficient test chemical for the expected duration of the trial. The mass of chemical will be recorded.

(2) Carrier gas flow (air or inert gas) will be established through the sparger or saturator cell, the heater will be turned on, and the apparatus will be allowed to stabilize at operating temperature. If a water bath is used, it is important to allow enough time for the chemical to equilibrate to the temperature of the water bath.

(3) The sparger or saturator cell will be operated at a slight positive pressure to deliver vapor at ambient pressure to the fixture. The sparger pressure will be measured using a mechanical pressure gauge with a range from 50 to 120 kilopascal (kPa). The sparger pressure with the fixture control software.

NOTE: The gauge must measure slightly above ambient sea level pressure to detect overpressurization, hence 120 kPa is required.

(4) After the trial, the remaining mass of chemical will be recorded.

(5) To prevent cross-contamination, a different sparger will be used for each test chemical.

c. Gas Challenge Generation.

(1) Gas challenges will be provided by disseminating from chemical gas cylinders into the mixing chamber of the fixture. Concentration can be controlled by adjusting the dilution of the challenge with air or nitrogen or via a mass flow controller.

(2) If the chemical is liquid inside a cylinder, a dual-flow valve may be used to inject inert gas into the cylinder headspace to eject liquid challenge compound.

4.4.4 Instrument Maintenance and Calibration.

a. Scheduled calibration and preventive maintenance will be performed on all instrumentation.

b. Installation, calibration, maintenance, and any instrumental failures will be documented. These may be documented in laboratory notebooks. Calibration records are considered part of the data package to be delivered to the customer.

c. Instruments should be calibrated and operated IAW Paragraph 3.4.1.

4.4.5 Fixture Component Integration Verification Procedures.

Before each trial, the test operator must verify that all calibrations are current and record the calibration date. Except for the flow rate instrumentation analysis, pretest calibration verifications are the same for small- and large-scale tests. These calibration verifications may be performed as described below:

a. All instrumentation must be verified at pretest and periodically during testing to minimize nonconformance if calibrated items drift. These verifications need to be documented in a laboratory notebook or other documentation.

b. Pretest Airflow Verification. Test apparatus flow measurements should be verified using the following process.

(1) A certified flow measurement device or primary flow reference (bubble meter, venturi, critical orifice, ASME-compliant flow nozzle, dry flow meter, laminar flow element, etc.) will be inserted in series with the flow meter being analyzed. Because of variation between the different apparatus types, the exact point of reference meter insertion will vary. Flow nozzles should be inspected regularly for corrosion or clogging.

(2) The verification should be done at a pressure and temperature as close as possible to the actual test conditions. Carrier gas must be clean dry air or nitrogen. The stream must be controlled by calibrated MFCs and measured using flow meters equipped with appropriate valves.

(3) Identifying information (manufacturer, serial number) and calibration data for the reference meter will be recorded.

(4) Airflow rate will be set to the target value and allowed to stabilize IAW applicable test methods.

(5) The room temperature and pressure will be recorded on the data sheet and used to reduce the data if volumetric or orifice flow controllers or meters are in use.

(6) The airflow rate will be measured/recorded using the flow controller being analyzed and the reference meter.

(7) Airflow rate measurement will be performed three times. The mean, standard deviation, and relative standard deviation (RSD) of the replicate flow measurements will be calculated for the flow meter being analyzed. The RSD should be less than 3 percent for small-scale tests and less than 5 percent for large-scale tests. Otherwise, corrective action should be taken that may include, but is not limited to, identifying and fixing a leak or cleaning the meter.

(8) The absolute deviation between the mean measured flow rate and the target flow rate will be calculated. If absolute deviation exceeds 5 percent of the target for small-scale tests or 10 percent of the target for large-scale tests, corrective action will be taken.

c. Other Sensor Verification.

(1) Temperature sensors must be verified by exposing them to a known temperature (e.g., room temperature or a liquid bath).

(2) Humidity sensors must be verified by exposure to at least two different NIST-traceable RH standards.

(3) Barometric pressure must be verified by comparison with local meteorology data or a calibrated barometer.

(4) against an independently calibrated manometer.

(5) GC detectors must be verified using a check shot with a known mass of chemical before each trial.

4.4.6 Pretest Fixture Verification.

a. Pretest fixture verification will include adjusting all temperature settings to the target value, establishing target humidity, and establishing the challenge concentration and airflow IAW the test plan.

b. Positive control verification trial(s) will be executed using the exact setup to be used with the APC. R123 [HCFC-123, 1,1-Dichloro-2,2,2-trifluoroethane, Chemical Abstracts Service® (CAS®) Number 306-83-2] or other approved non-absorbed unreactive vapor must be used to ensure that air is not leaking around the APC. This leak test must be performed before verification trials. If each trial result is within the tolerance of the target value (Paragraph 2.3), the system will be considered to be operating properly. If each result is not within this range, the test apparatus will need analysis and modification before running verification trials. If the test plan requires simultaneous testing of more than one test item, then a test item must be affixed to each outlet during verification trials. Verification trials must be completed and analyzed before trials of record are performed.

NOTE: Some of the positive verification trials may be used as the pilot trials(s) and will be conducted IAW Paragraph 4.5.

4.4.7 Test Validation (Pilot Trial).

The pilot trial will be conducted IAW the procedures described in Paragraph 4.5 using an APC whose performance has previously been validated.

NOTE: The pilot trial is not a substitute for a complete V&V of the fixture (Paragraph 7.1). The pilot trial augments the V&V and ensures that the fixture is ready to conduct the test as defined in the test plan. The fixture V&V must be completed before beginning the pilot trial.

a. A validation trial for each chemical should be conducted after fixture verification and before every major test, or series of trials. Published test reports may contain applicable validation data[3,24,25,26].

b. The breakthrough time for each test chemical should be within 10 percent of values from previously accepted trials. If sufficient data are available, results should be compared to control charts to see if the fixture is performing within control limits.

4.5 APC Test Procedures.

Tests the four types of APCs, which use similar procedures (Paragraph 4.5.1). Procedures specific to the type of testing (filtration media, FFs, IP canisters, and CP canisters) are outlined in Paragraphs 4.5.2 through 4.5.6. Many procedures will be specific to a particular test item and must be described in the test plan.

4.5.1 General APC Test Procedures.

a. If BFC exposure is required, filtration media, canisters, and FFs should be exposed before the chemical vapor challenge (Appendix B). The weight of each APC should be recorded before and after BFC exposure.

b. The APC will be stored in a sealed container until needed for preconditioning (if applicable) or testing.

c. The APC must be weighed before being placed in the test fixture. The APC will be installed in the test fixture and then checked for proper installation. The APC must be installed so that the challenge gas cannot leak out of the test fixture without passing through the APC. If O-rings are used for APC installation, a seal test will be performed using methods specific to the type of APC. If the seal test fails, the APC will be removed, examined, replaced as needed, reinstalled, and retested until it passes.

d. All control systems and automatic data collection/recording software systems will be checked for readiness. All instrument clocks should be synchronized if separate clocks are being used on various instruments. Environmental control parameters will be adjusted IAW the test requirements. A programming system such as LabVIEW® (National Instruments, Austin, Texas) will be used to control and monitor environmental conditions and to record operational conditions.

e. Environmentally conditioned air will be directed through the APC to precondition the APC to the trial conditions. Effluent temperature and humidity must be within the test control tolerances before preconditioning ends (Paragraph 2.3).

f. Barometric pressure will be measured at least one time for each trial. It may be convenient to use data recorded at a site within 10 km of the laboratory, so that the test team need not buy and calibrate a barometric pressure transducer. Pressure transducers used with continuous infrared analyzers are required to be calibrated.

g. In preparation for the trial, the challenge airflow should be started in bypass mode (Appendix A). Temperature and humidity should be stabilized first IAW the DQOs (Paragraph 3.4.3.a). Once the environmental conditions are stable, the dissemination can start. Before the trial is initiated, at least one reading of effluent concentration data will be collected to characterize the background concentration for the vapor challenge. These data will be used to correct the trial effluent concentration data (Paragraph 6.1).

h. Once the APC is preconditioned, the challenge airflow will be diverted through the APC to start the trial. The challenge concentration will be controlled and recorded. The humidity and temperature must be recorded and may be controlled. F FF monitored instead of airflow.

i. The effluent concentration and airflow must be monitored and recorded. Monitoring the temperature and humidity of the effluent is optional. Monitoring of effluent humidity provides additional information on the effect of challenge humidity on test item filtration performance.

j. The trial will be terminated when the effluent concentration reaches the breakthrough concentration (specified by the test plan), when the test item has received the target concentration × time (Ct), or when a predetermined trial duration has been reached, or other trial end criterion defined in the test plan. To terminate the trial, the challenge airflow will be diverted to sacrificial filters or scrubbers. The dissemination of challenge chemical and humidity will cease. The time of termination is the trial stop time.

k. When a desorption test is required according to the Test and Evaluation Master Plan or other requirements document, data will continue to be recorded after the dissemination has ceased. Chemical-free process air will be allowed to continue to pass through the test media or filter. The trial will be terminated when the effluent concentration decreases below the predetermined level or when a predetermined time limit has been reached (specified by the customer).

NOTE: While the APC is air washed, any challenge chemical not permanently bound to the media or filter will desorb.

l. The APC will be removed and weighed. The APC will be discarded IAW the test center's waste handling regulations.

m. To prepare the fixture for the next trial, an air wash of the fixture may be performed with air heated to the highest temperature at which the fixture is validated. Other steps may be taken to decrease the background concentration before the next trial. Steps may include a solvent rinse, a flush with cleaning vapor, or replacement of fixture components. If fixture components need to be replaced, care must be taken not to change the baseline configuration of the fixture.

n. Preparation for the Next Test Chemical.

(1) For Vapor Challenge Testing.

(a) Used syringes will be cleaned with an applicable solvent. If the plunger still moves freely, there are no deposits from the previous test compound, there is little carryover, and the syringe may be used for the next test compound. Otherwise, the syringe will be discarded and replaced.

(b) Used diffusion or permeation tubes will be replaced.

(2) For Gas Challenge Testing. Nonempty cylinders will be replaced with cylinders containing the new chemical. Empty cylinders will be returned to the vendor or discarded.

o. Calibration of test instruments will be verified. Out-of-calibration test instruments will be replaced and preventive maintenance will be performed.

4.5.2 Small-Scale Filtration Media Test.

a. Small-scale filtration testing will be conducted as described in Paragraph 4.5.1 using the small-scale filtration media fixture (Figures A.2 and A.3).

b. Filtration Media Preparation.

(1) The desired mass of media will be calculated based on the computed target bed volume and the density of the media.

(2) The bulk density of the filtration media (as packed) will be determined by storm filling [Paragraph 4.5.2.b(6)] a sufficient volume of media into a graduated cylinder IAW the American Society for Testing and Materials (ASTM) International (West Conshohocken, Pennsylvania) D2854-96[27] and then weighing the cylinder. The density determination should be replicated at least three times.

(3) The filter media (usually granules or beads) or material being tested is normally provided in a jar or other sealed container. The filter media should be thoroughly mixed so that a uniform sample of material may be withdrawn. A vee blender is recommended for mixing the filter media; however, other types of equipment may be used.

(4) In some cases, the supplier will mix the material before shipping, and additional mixing may not be required. The contents of the supplied material should be inspected for any visible separation by size, color, or other media property.

(5) Filtration media sample will be weighed in a container. The weight and volume of the media will be recorded on the data sheet.

(6) Test Cell Media Loading.

After the media have been mixed thoroughly, the mass of granular test media needed to achieve the target bed volume will be calculated. Test cells will be loaded with media using the apparatus described in Appendix A, Paragraph A.2.e.

(a) The test cell (Figure A.3) should be positioned vertically and the bed support should be perpendicular to the test cell and level.

(b) The storm filler (Figure A.6) will be placed on top of the test cell and aligned vertically and coaxially. The bed-leveling device will be used to level the top of the packed bed when the storm filling operation is completed. The bed-leveling device is constructed of an inert material and designed to fit closely into the test cell.

(c) A feeder (Figure A.6) will be positioned so that the granules will fall into the storm filler.

(d) The mass of granular test media will be measured to ensure the targeted bed volume has been achieved.

(e) The test cell will be filled by pouring the granular test media into the reservoir of the storm filler. The feeding rate should be less than 4.0 cm/min. Therefore, the feed rate will be analyzed by recording the time it took to fill the test cell. If the feed rate exceeds 4.0 cm/min, the media should be removed from the test cell, and the test cell will then be refilled at the reduced feed rate.

(f) For multilayer media beds, the filling procedures will be repeated beginning with the downstream media and working upstream with each media.

(g) After the full mass of media has been loaded into the test cell, the storm filling apparatus will be removed from the test cell.

(h) Using the bed-leveling device, the top of the test cell bed will be leveled (Figure A.6). The Teflon® (DuPont, Wilmington, Delaware) leveling device will be gently placed within the cell so that it will make contact with the top of the packed bed. Care should be taken not to compress the bed and skew results. The bed-leveling device will be removed slowly so the bed will not be disturbed.

(i) The bed will be measured with a ruler to verify the target bed depth is achieved and then transferred to the test cell.

4.5.3 FF Test.

FF testing will be conducted as described in Paragraph 4.5.1 using the SWIFT fixture (Figures A.5 through A.7). There are two additional procedures required for FF testing. Firstly, swatches will be prepared and installed in test cells (Paragraph 4.5.3.1). Secondly, the usually controlled instead of airflow rate (Paragraph 4.5.3.2).

4.5.3.1 Preparation of Swatches for FF Trials.

a. FF swatches will be cut from the square panels and weighed. Each swatch will have a 5.1-cm diameter and can be up to 1 cm thick.

b. The shelter-exterior side of the swatch will be the challenge side while the shelter-interior side of the swatch will be the effluent measurement side. The extra ports on the cup will be plugged and then a cap connected to a pressurized air source will be screwed into the agent challenge port in the top. Each swatch will be mounted in the swatch cup using O-rings (Figure A.9).

c. After each swatch is mounted in a test cup, a seal test will be conducted to verify that the O-rings are sealed correctly (TOP 08-2-501[28]). Each cup will be pressurized at 3.0 iwg and then the air source shut off. Each cup is required to hold a pressure of at least 2.9 iwg for 30 seconds or a pressure of at least 2.0 iwg for 2 minutes.

d. If the seal test fails, the cup will be disassembled, examined, repaired as needed, reassembled, and retested until the cup holds pressure. The seal test results will be recorded.

e. After passing the seal test, each cup will be inserted into the SWIFT Fixture (Figure A.9).

4.5.3.2 C .

The airflow rate and ΔP through each filter will be monitored.
controlled instead of airflow rate. any adjustments of the airflow rate to reach and maintain the target ΔP will be recorded. Automatic or manual adjustment may be used to sustain a constant ΔP accounting for test item variation or loading.

4.5.4 IP Canister Test Procedure.

IP canister testing will be conducted IAW Paragraph 4.5.1 except that each IP canister will be weighed and then installed using an adapter in the fixture (Figure A.7). The high-mass flow rate of test chemical requires the use of a sparger for dissemination of vapor. The high-mass flow rate will require the use of a CP canister as a sacrificial filter for all chemical challenges. No seal test will be performed.

4.5.5 CP Canister Test Procedure.

CP filter testing will be conducted IAW Paragraph 4.5.1 except that each CP filter will be weighed and then installed in the AAPTF (Figures A.11 and A.12). The high-mass flow rate of test chemical requires the use of a sparger for dissemination of vapor. The high-mass flow rate will require the use of a building-scale sacrificial filter for all chemical challenges. The CP canisters require the following procedures additional to those in Paragraph 4.5.1 for installation, seal testing, and removal from the test fixture.

a. The outer filter housing plate will be secured in place and bolted. The filter housing will be customized to fit the CP canister. A customized CP filter adapter may be required to fit various sizes of CP canisters.

b. Seal testing will be performed to ensure proper seating of the CP canister in the filter housing and to analyze the integrity of the filter housing's vapor containment. The seal test of the CP canister will be conducted in place within the filter housing using halide (DPG SOP DP-0000-U-105[29]), Vertrel® (ECBC SOPs RNG-035[30] and RNG-237[31]) or similar method. The halide test compares detector measurements taken upstream from the canister with downstream measurements. The halide test must show at least a 3-log reduction of concentration downstream.

4.5.6 CP Fabric Test Procedure.

a. CP fabric testing will be conducted IAW Paragraph 4.5.1 with the following exceptions:

(1) Each CP fabric will be weighed and then installed in an adapter within the filter housing of the AAPTF (Figures A.11 and A.12).

(2) during this type of testing.

b. The filter housing will be customized to fit the CP fabric. A customized CP fabric frame adapter may be required to fit various sizes of CP fabric panels.

c. Seal test for fabrics should be conducted IAW Paragraph 4.5.5.b.

d. CP fabric will be air washed after the trial, removed, and then immersed in suitable decontaminant solution.

5. DATA REQUIRED.

a. A written summary of the receipt inspection will be added to the test report and/or a test incident report. Test and referee data collected and processed will be stored in a test data-management system within the testing agency.

b. A complete daily electronic or paper log with a detailed description of each trial will include the following: APC TICN, target challenge vapor concentration, target temperature, target humidity, identification and purity of test chemical, calibration data package for all

instrumentation specifying the range over which each instrument was calibrated, and critical system performance instrumental parameter logs.

c. Data measurements performed once per trial include the following:

(1) Preconditioning start and stop times

(2) Vapor challenge start and stop times.

(3) Trial start and stop times.

(4) Trial duration.

(5) Mass of test chemical loaded.

(6) APC weight before BFC exposure.

(7) APC weight after BFC exposure

(8) APC weight immediately before environmental conditioning

(9) Inlet airflow.

(10) Dissemination rate

(11) Temperature of the dissemination system (if applicable).

(12) APC weight after exposure to chemical vapor.

(13) Other vapor generation and dissemination settings used.

d. The environmental measurements will include measured humidity, temperature, C The measurement interval will not exceed 1 minute. These measurements will be plotted.

e. The chemical concentration measurements will include challenge concentration and effluent concentration. The measurement interval will not exceed 15 minutes.

f. Calculated values will include challenge Ct, mass of test chemical disseminated (Ct × airflow rate), breakthrough time for each APC, and the mass of chemical consumed. If the breakthrough concentration criterion is undefined, the concentration will be set at the 10-minute upper marginal MEG level as defined by the former U.S. Army Center for Health Promotion and Preventive Medicine[22] (USACHPPM), now known as the Army Public Health Center (Provisional).

g. Test fixture configuration data will include drawings, specifications, photographs, software, and V&V data.

h. Any issues encountered will be recorded in test incident reports if applicable.

6. PRESENTATION OF DATA.

Data architecture must be defined to deliver traceable data flow through the testing process. Results and interpretation must correspond to the test objectives and be presented in the final test report.

6.1 Data Processing.

a. Background concentration is the concentration measured by instrumentation before each trial and is caused by off-gassing of residual contamination. Measured effluent concentration is the sum of the background concentration and the true effluent concentration. To obtain the true effluent concentration, the background concentration should be subtracted from the measured value.

b. Mathematical background correction should only be used when operational efforts cannot reduce the background level below DQOs.

(1) When background levels are observed above this criterion, the effluent concentration should be corrected mathematically using the most applicable option.

(2) If pretrial readings show decay in the fixture off-gassing that is approximately exponential, then an exponential curve will be fitted to concentration data collected during the 30 to 60 minutes before trial start. The extrapolated concentrations will be subtracted from the effluent readings.

(3) If pretrial readings are approximately constant, then the average for concentrations measured during the 30 to 60 minutes before trial start will be calculated and subtracted from the effluent readings.

(4) If there are no pretrial readings but the early trial readings show a clear fixture off-gassing decay followed by breakthrough much later (i.e., curve passes through a minimum), then an exponential curve will be fitted to the off-gassing portion of the data. The curve will be subtracted from the readings taken during the trial period. This option will be used for data where breakthrough was not simultaneously taking place with the early fixture off-gassing.

c. Breakthrough time is the elapsed time when the true effluent data reaches the MEG value[22] or other value specified by the customer. MEG values are of two types: threshold concentration and concentration time-weighted average (TWA). The default breakthrough concentration is the upper marginal MEG level[22], unless otherwise specified in the test plan. The breakthrough time is calculated by interpolation of the effluent concentration or TWA at the MEG value.

(1) The MEG value used for simulant will be the agent value. For instance, the MEG for sarin (GB) will be used for the GB simulant.

(2) For the 24-hour, 8-hour, and 1-hour MEGs, the breakthrough can be defined using a trailing TWA (Equation 5). The TWA is determined over a sliding time window trailing the time point it represents (e.g., the 1-hour TWA is the average concentration over the last hour).

$$\mathrm{TWA}_x(t) = \frac{1}{60x}\int_{t-60x}^{t} c(t')dt' \qquad \text{Equation 5}$$

Where,

TWA_x = the x hour time weighted average of challenge concentration in mg/sm³
x = the length of the TWA in hours (i.e., 1 hour, 8 hours, or 24 hours).
t = the elapsed time in minutes.
$c(t)$ = the challenge concentration in mg/sm³.

NOTE: c was zero before $t = 0$ because there were no background-corrected data before the start of the trial. t' = the variable of integration.

d. Challenge Ct of each trial must be calculated using Equation 6. Desorption is measured after challenge and does not affect Ct.

$$Ct = \int_0^{t_D} c(t)dt \qquad \text{Equation 6}$$

Where,

Ct = the exposure in mg min/am³
$c(t)$ = challenge concentration as a function of time in mg/am³
t = the elapsed time of the trial in minutes
t_D = the trial duration in minutes

e. The mean challenge concentration will be calculated using Equation 7. Total mass of the challenge may be calculated using Equation 8.

$$\bar{c} = \frac{Ct}{t_D}$$ Equation 7

Where,

$\bar{c}$ = the mean concentration in mg/am³

Ct = the challenge in mg min/am³ (Equation 5)

t_D = the trial duration in minutes

$$m = \bar{c}\,V$$ Equation 8

Where,

m = the challenge mass in mg

$\bar{c}$ = the mean challenge concentration in mg/am³ (Equation 6)

V = the total volume of air that has flowed through the APC in am³

f. It is often desirable to compare the collected data with reference data (historical test results). The experimental breakthrough time can be normalized to represent the breakthrough time at exactly the reference concentration (Equation 9). Data normalization may only be performed if the reference value is within 20 percent of the actual average challenge concentration.

$$t' = t\frac{\bar{c_a}}{\bar{c_r}}$$ Equation 9

Where,

t' = the breakthrough time normalized for challenge variation in minutes

t = the measured breakthrough time in minutes

$\bar{c_a}$ = the average challenge concentration measured during the test trial in mg/am³

$\bar{c_r}$ = the reference challenge concentration in mg/am³

g. Elapsed time may be corrected to account for analysis time and transit time (time it takes for the test chemical to reach the instrument). Some analytical techniques, particularly for low effluent concentrations (e.g., pre-concentration techniques), inherently have a time delay between obtaining a sample and reporting the concentration of that sample, particularly if one instrument is analyzing multiple samples. The correction for transit time is the last term in

Equation 10. The need for this correction can be reduced by minimizing the length and diameter of tubing. Equation 10 corrects the elapsed time for both transit time and analysis time. These corrections should be applied if they exceed the uncertainty of the breakthrough time.

$$t_c = t' - t_a - \frac{V_t}{Q} \qquad \text{Equation 10}$$

Where,

t' = the breakthrough time normalized for challenge variation from Equation 9 (minutes)

t_c = the corrected elapsed time (minutes).

V_t = the tubing volume (aL).

Q = the flow rate in aLpm.

t_a = the analysis time delay (minutes).

h. In some instances a customer may wish to have the effluent Ct calculated. In this case, it can be calculated using Equation 6, but using effluent concentration readings instead of challenge concentration readings.

i. Breakthrough Time Correction for Weakly Adsorbing Gas.

(1) A challenge chemical that elutes from the test media or filter following termination of dissemination is a chemical that has not been irreversibly retained or removed by the media or filter.

(2) Normalized breakthrough time is corrected using Equation 11.

$$\Delta t = \frac{Ct - Ct_d}{\bar{c}} \qquad \text{Equation 11}$$

Where,

t = the breakthrough time correction in minutes.

Ct = the exposure of chemical challenge in mg min/am³ (Equation 5).

Ct_d = the exposure due to chemical that desorbed from the media or filter in mg min/am³, computed by numerically integrating the effluent concentration following challenge chemical termination in a manner similar to that shown in Equation 5.

$\bar{c}$ = the mean measured concentration of chemical in the challenge stream in mg/am³ (Equation 6).

j. Determination of Accuracy.

Accuracy is the measure of how close to the actual value a measurement is on average. The metric for accuracy is the mean relative percent difference (Equation 12). Best practice requires at least three measurements, but seven are recommended.

$$\overline{\text{RPD}} = \frac{100}{N x_a} \sum_{i=1}^{N} |x_i - x_a| \qquad \text{Equation 12}$$

Where,

$\overline{\text{RPD}}$ = the mean relative percent difference (RPD)
N = number of measurements taken during calibration
x_i = the measurements taken during calibration
x_a = the value of the standard

k. Determination of Precision.

Precision is the measure of variation in the measurement. The metric for precision is the RSD (Equations 13 and 14). Best practice requires at least three measurements, but seven are recommended.

$$s = \sqrt{\frac{1}{n-1} \sum_{i=1}^{n} (x_i - \bar{x})^2} \qquad \text{Equation 13}$$

Where,

s = standard deviation with n - 1 degrees of freedom
n = total number of observed values
$\bar{x}$ = mean of observed values.
x_i = each measurement.

$$\text{RSD} = \frac{100s}{\bar{x}} \qquad \text{Equation 14}$$

Where,

RSD = the relative standard deviation expressed as a percent.
s = standard deviation with n - 1 degrees of freedom
$\bar{x}$ = mean of observed values.

l. Uncertainty.

(1) The standard reference for calculation of uncertainty is documented in the *Guide to the Expression of Uncertainty in Measurement* (GUM)[21]. The approach used in this procedure is considered a statistical evaluation of uncertainty by analyzing data (i.e., a Type A evaluation in GUM terminology). Uncertainty is determined by combining test measurement uncertainty with an evaluation of other sources of uncertainty. For many instruments, the uncertainty of the instrument is provided with the documentation.

(2) Determine the test measurement uncertainty from test data using Equation 15.

$$U = t_{95}\frac{RSD}{n} \qquad \text{Equation 15}$$

Where,

U = uncertainty relative to the mean.
t_{95} = Student's t distribution value for n-1 degrees of freedom at the 95 percent confidence interval.
RSD = relative standard deviation (Equation 13).
n = number of samples analyzed (minimum of 3 samples, but 7 is recommended).

(3) The uncertainty for each measurement will be determined by combining the individual uncertainties from each source, using sum of squares (Equation 16). Sources of uncertainty include test measurement, calibration, test method, analytical solutions, sampling, test chemical, water vapor, and heat transport within the fixture, storage and handling of test chemical solutions and APCs, and test chemical purity. Uncertainty values derived from documentation should be used when possible. The overall uncertainty for breakthrough time is the combined uncertainties of all applicable measurements (Equation 16).

NOTE: The calculation of uncertainty may not significantly change after the highest three or four sources of uncertainty are included.

$$U_m = \sqrt{\sum U_i^2} \qquad \text{Equation 16}$$

Where,

U_m = the total uncertainty.

U_i = the uncertainty from each source.

6.2 Data Presentation.

a. Test reports must contain all the data necessary to demonstrate that the test item was challenged correctly. The required information includes an analysis of uncertainty in the test data. Test documents must state explicitly which units are used.

b. The final test report must contain all the data necessary to analyze the performance of the APC. However, the test report should not contain all data collected. Some types of data may be useful to the customer or other members of the testing community without being necessary to the evaluation of the APC. These useful, but not necessary, data may be compiled into a supplementary data package. The supplementary data package can be provided on a digital video disc (DVD) or compact disc (CD) upon request or stored on a website such as Versatile Information Systems Integrated ON-line (VISION) Digital Library System (VDLS).

6.2.1 Trial Conditions.

For each trial:

a. Target and mean for challenge concentration, temperature, humidity, barometric pressure, achieved challenge Ct, time to achieve target challenge Ct, and trial duration will be tabulated in the data package and in the report (Table 1).

b. Environmental and challenge conditions will be plotted showing the required tolerance for each parameter in the data package (e.g., Figure 2). Selected plots will be published in the report.

6.2.2 APC Results.

For each APC:

a. Effluent concentration vs. time plots and tables will be provided in the data package (e.g., Figures 3 through 6). Selected plots and tables will be included in the report.

b. 2) in the data package and the report.

c. APC weight change and breakthrough time with means and standard deviations will be tabulated (e.g., Table 3) in the data package and the report. If any corrections are performed, corrected and uncorrected values will be presented.

d. Breakthrough time will be plotted for comparison (e.g., Figure 7) in the data package and the report. If any corrections are performed, corrected and uncorrected values will be presented.

6.2.3 Uncertainty.

The following will be placed in the data package and report:

a. The accuracy and precision will be tabulated as a QC measure to characterize the effectiveness of the measurement system (Tables 4 and 5).

b. The uncertainty from each source will be tabulated (Table 6). A table for each measured parameter is required.

c. The total uncertainty of effluent concentration, challenge concentration, environmental conditions, and breakthrough time will be tabulated (Table 7).

TABLE 1. EXAMPLE ENVIRONMENTAL AND CHALLENGE CONDITIONS FOR EACH TRIAL.

Trial Name	Compound[a]	BFC[b]	Temperature (°C)		RH[c] (%)		Concentration (mg/am³)[d]		Time to Target Ct[e] (hour)	Duration (hour)	Barometric Pressure (mbar)
			Target	Mean	Target	Mean	Target	Mean			
1	GB	Fog oil	12	15	48	49	1000	928	0.09	3.0	875.84
2	TMP	Fog oil	5	8	0	5	300	291	0.28	18.8	865.62
3	TMP	None	25	25	70	75	300	315	0.25	11.1	868.48
4	TMP	Fog oil	25	25	70	74	1000	923	0.09	16.4	869.73
5	TMP	None	55	51	0	1	1000	1068	0.08	4.0	865.42
6	GB	None	18	20	70	70	300	292	0.27	8.0	868.88
7	TMP	JP-8[f] exhaust	22	22	31	36	300	319	0.25	23.0	869.53

[a]GB – sarin; TMP – trimethyl phosphate.
[b]Battlefield contaminant.
[c]Relative humidity.
[d]Concentration in milligrams per actual cubic meter of air at ambient conditions.
[e]Concentration × time.
[f]Jet propellant fuel, type 8.

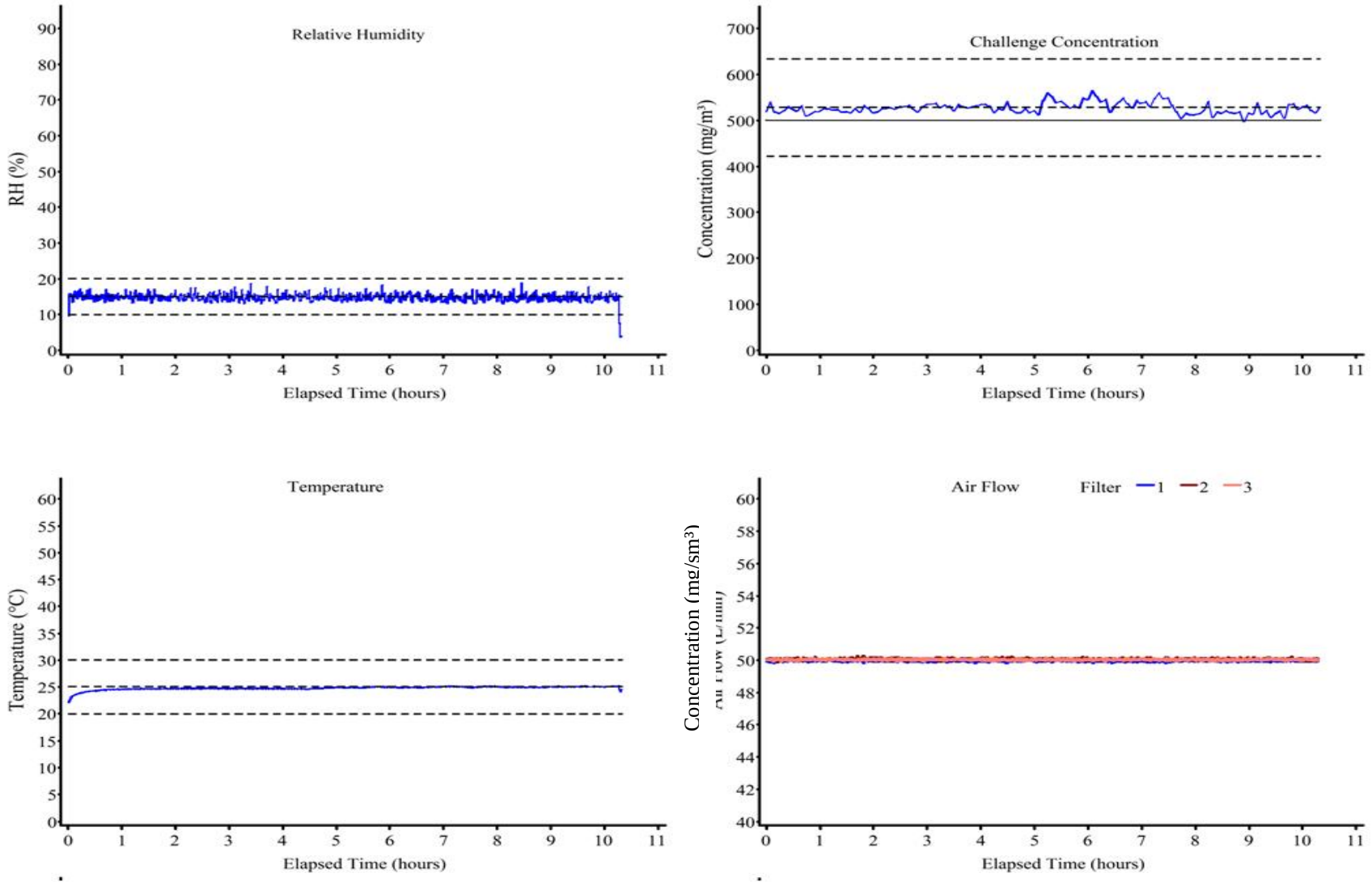
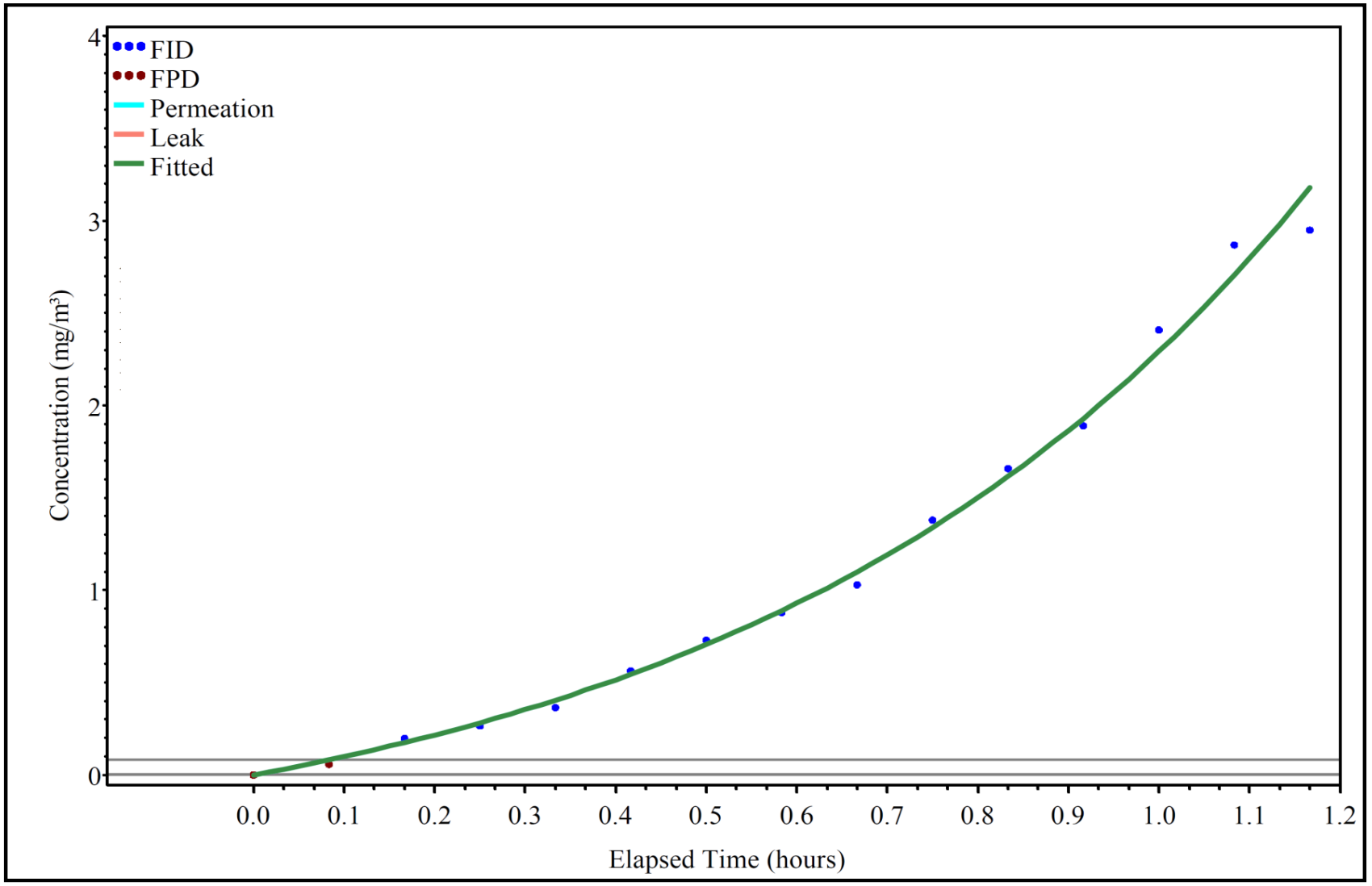


Figure 2. Example environmental and challenge conditions.



NOTE: FID – flame ionization detector; FPD – flame photometric detector.

Figure 3. Example effluent concentrations for a filter fabric (FF) test.

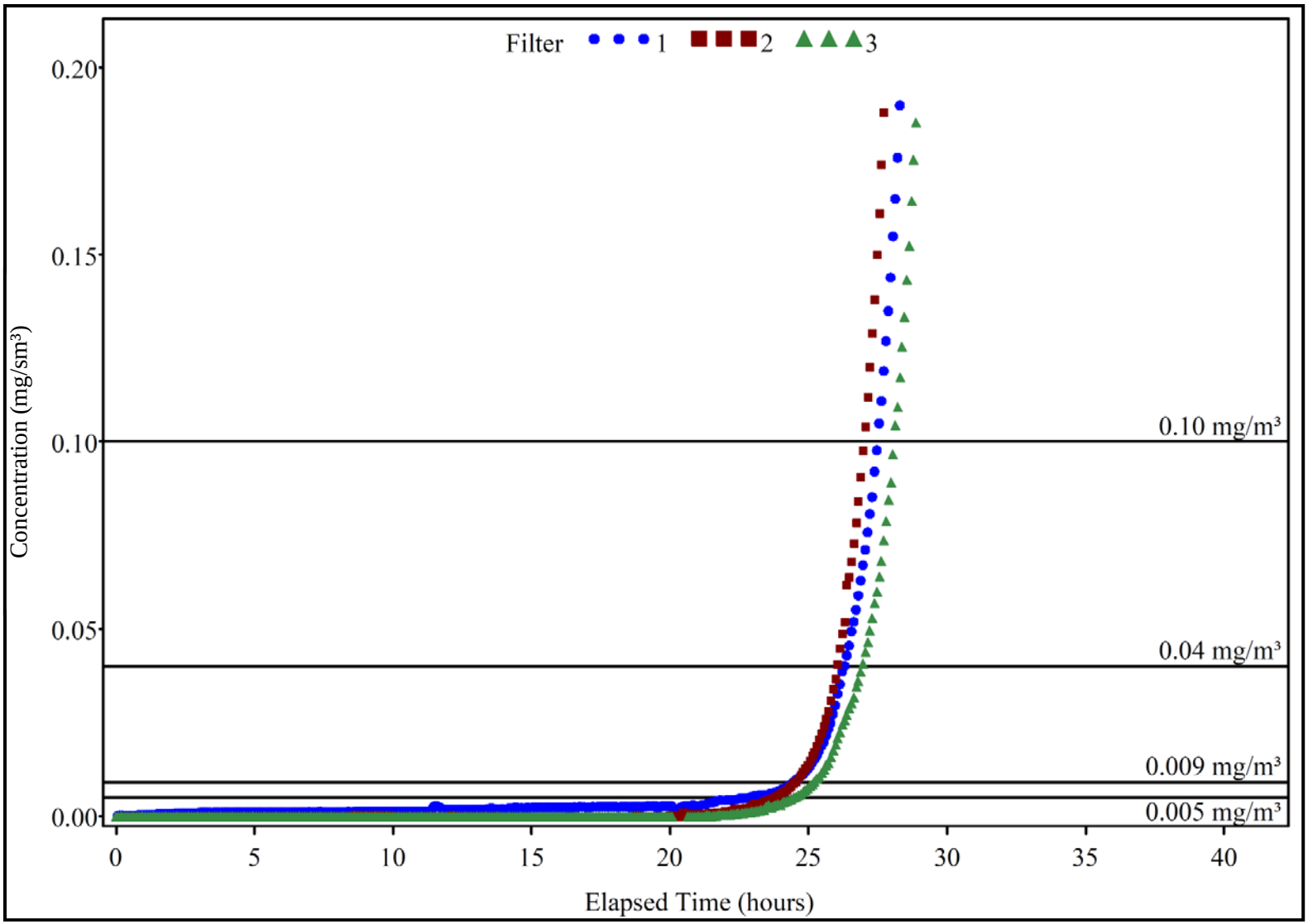
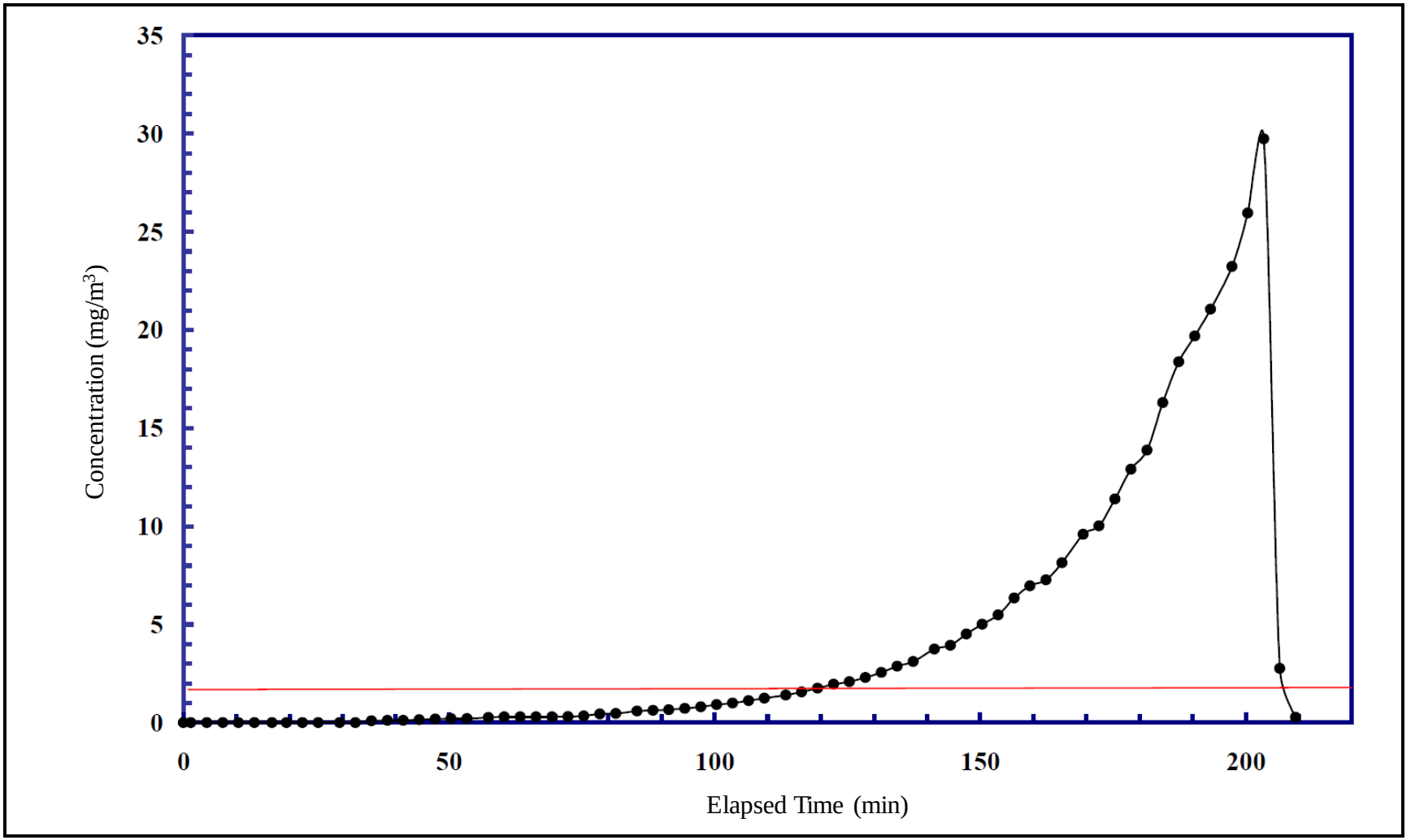


Figure 4. Example effluent concentrations for an individual protection (IP) canister test.



NOTE: The red line is the lower calibration limit.

Figure 5. Example effluent concentrations for a small-scale filtration media test plotted against the elapsed time.

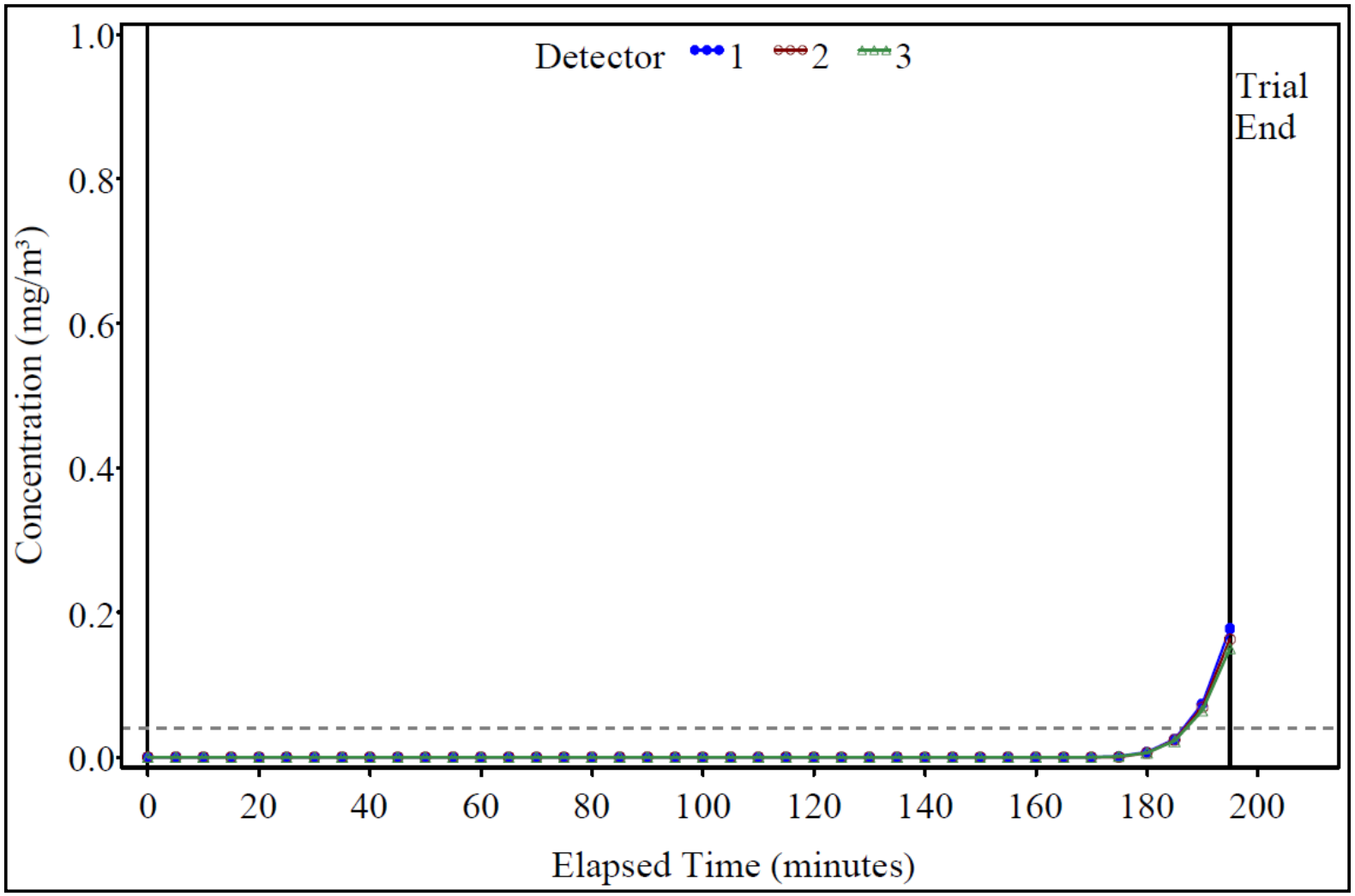


Figure 6. Example of effluent concentrations from a collective protection (CP) canister test plotted against the elapsed time.

TABLE 2. E E FFEREN RESS RE N RF F R E C R R F C N C NEN (APC) DURING A TEST.

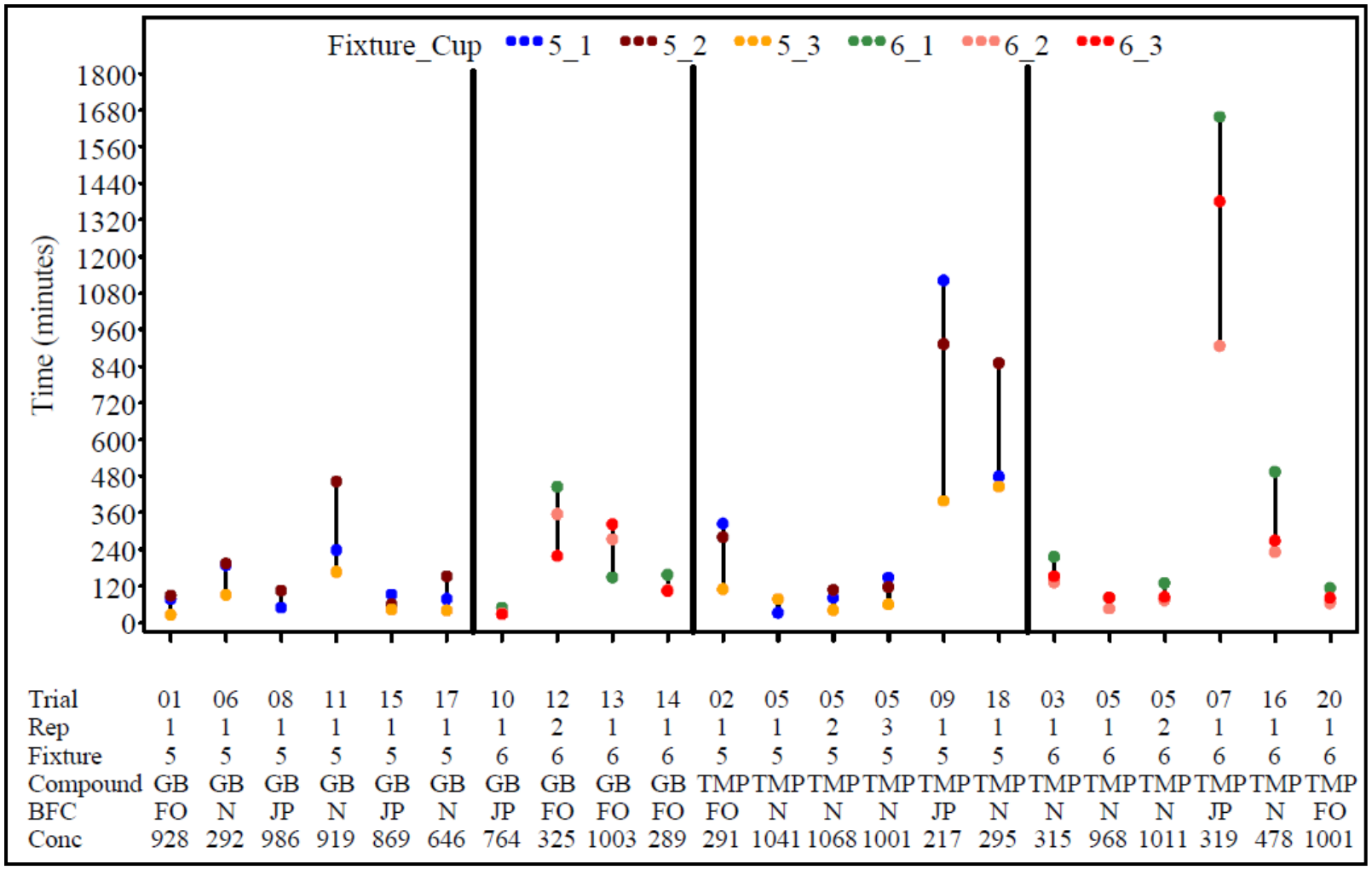
Repetition	Fixture	APC TICN[a]	(iwg[b])	Mean (iwg)	SD[c] (iwg)	Mean Air Flow (aLpm[d])
1	5	A32	0.10	0.10	0.01	0.91
	5	A33	0.10	0.10	0.01	0.87
	5	A34	0.10	0.10	0.01	0.85
2	6	A35	0.10	0.10	0.01	0.74
	6	A36	0.10	0.10	0.01	0.84
	6	A37	0.10	0.10	0.01	0.75

[a]Test item control number.
[b]Inches water gauge.
[c]Standard deviation.
[d]Actual liters per minute.

TABLE 3. EXAMPLE BREAKTHROUGH TIME AND WEIGHT CHANGE FOR EACH AIR PURIFICATION COMPONENT (APC) DURING A TEST.

Repetition	Fixture	APC TICN[a]	Breakthrough Time (hours)	Mean (hours)	Standard Deviation (hours)	Weight Change (g)	Mean	Standard Deviation
1	5	A32	5.2	6.8	1.8	21.4	22.3	10.7
	5	A33	6.3			33.4		
	5	A34	8.8			12.1		
2	6	A35	9.2	7.7	1.4	21.7	18.0	5.8
	6	A36	6.5			20.9		
	6	A37	7.4			11.3		

[a]Test item control number.



NOTE: Rep – repetition; GB – sarin; TMP – trimethyl phosphate; BFC – battlefield contaminant; FO – fog oil; N – none; JP – jet propellant fuel, type 8 (JP-8) exhaust; Conc – the mean challenge concentration in mg/am³.

Figure 7. Example time to breakthrough at the military exposure guideline (MEG) level plot.

TABLE 4. EXAMPLE ACCURACY OF TEMPERATURE AND RELATIVE HUMIDITY (RH) FOR AGENT VAPOR TRIALS.

Number of Trials	Absolute Difference Between Target and Actual Mean Temperature (°C)		Absolute Difference Between Target and Actual Mean RH (%)	
	Mean	90th Percentile	Mean	90th Percentile
32 (15[a])	0.86	1.3	4.23	8.8

[a]Of the 32 trials conducted with controlled temperature, 15 also had controlled RH.

TABLE 5. EXAMPLE PRECISION OF TEMPERATURE AND RELATIVE HUMIDITY (RH) FOR AGENT VAPOR TRIALS.

Number of Trials	Standard Deviation of Difference Between Target and Actual Mean			
	Temperature (°C)		RH (%)	
	Mean	90th Percentile	Mean	90th Percentile
32 (15[a])	0.30	0.6	1.19	2.4

[a]Of the 32 trials conducted with controlled temperature, 15 also had controlled RH.

TABLE 6. EXAMPLE PARAMETER (TEMPERATURE) AND COMBINED UNCERTAINTY.

Source	Accuracy (°C)	Precision (%)
Calibration	0.113	0.46
Measurement	0.86	1.0
Placement	0.6	Not applicable
Combined Uncertainty	1.1	Not applicable

TABLE 7. EXAMPLE OF TOTAL UNCERTAINTY IN TEST MEASUREMENTS.

Source	Uncertainty (%)	Reference
Temperature	1.1	Table 6
Relative humidity (RH)	2.3	Paragraph 2.2
Flow rate	5.0	Paragraph 2.2
Challenge concentration	10.0	Paragraph 2.2
Effluent concentration	20.0	Paragraph 2.2
Combined Uncertainty	23.1	Equation 16

7. V&V.

Per ATEC Regulation 73-21[32], all test fixtures, models, and simulations to include simulant selection process must be verified and validated. Any V&V test plan and results will need to be reviewed and accepted by the relevant CAPAT [Office of the Deputy Under Secretary of the Army, Test and Evaluation (DUSA TE)]. Accreditation of fixtures, models, and simulants will be at the discretion of each operational test agency (OTA). For a joint program, accreditation will be coordinated through the lead OTA.

NOTE: The test fixture is validated with a particular method, for a particular APC and specific agents/simulants. The test fixture, test and analytical method, as well as simulant selection V&V may be completed simultaneously.

7.1 APC Test Fixture V&V.

If an adequate test fixture for a specific test does not already exist, a test fixture will be constructed IAW drawings and specifications stored in the organization's configuration management system. The test fixture then must undergo V&V per the following procedures:

a. All V&V testing must show that the fixture will perform to the specifications outlined in this TOP and appropriate test and evaluation capabilities and needs (TECN) guidance published by the CAPAT.

b. Test fixture V&V is required to ensure that the test fixture can control and maintain the required test conditions. V&V test parameters include airflow rate, dissemination flow rate, challenge concentration, challenge air inlet temperature, and RH or WVC. Lack of consistency in these parameters will affect the performance measurement. Each parameter under test control in the fixture must be maintained within the permissible errors of measurement and tolerance ranges specified in Paragraphs 2.2 and 2.3.

(1) The performance of the entire test fixture can be verified by testing a previously tested test item and comparing current test breakthrough times with the established or expected performance norms.

(2) If no performance norms have been established for the chemical, filter media, and filter design, then trials may be verified by comparison with predicted breakthrough times. Use of the MultiVapor[33] model (from NIOSH) is the recommended method of predicting APC performance[34].

(3) Verification trials should be performed using the procedures from Paragraph 4.5. The fixture should be verified across the range of environmental conditions required by the test. If no range is given, at least one verification trial should be performed at ambient temperature and humidity. DoE should be used to cover the bounds of operational performance and be documented in the V&V plan.

7.2 Analytical Method V&V.

Analytical test methodology for new test chemicals and BFCs should be developed and validated before test execution is scheduled to begin. Precision, accuracy, repeatability, and reproducibility should be determined. Repeatability is the standard deviation between successive trials with the same operator on the same fixture. Reproducibility is the standard deviation between successive trials on different fixtures. A new method requires pilot trial(s) before test execution.

7.3 Simulant Selection V&V.

a. Simulants must be verified and validated before being used in APC testing. Selection of simulants should be conducted IAW TOP 08-2-196[9]. The objective will be framed, potential simulants will be identified and screened, simulants that best mimic the desired agents will be selected, and the selected simulants will be verified and validated. For APC testing, selected simulants need to mimic the adsorptive or reactive behavior of an agent. APC tests may require the use of a novel simulant to mimic particular characteristics of the agents of interest.

b. The following list represents simulant materials that have been verified, validated, and used in place of adsorptive agents: trimethyl phosphate (TMP, CAS® Number 512-56-1); triethyl phosphate (TEP), CAS® Number 78-40-0; and tripropyl phosphate (TPP), CAS® Number 513-08-6[1,24,25,35,36].

c. The following list represents simulant materials that have been used in place of adsorptive agents but have not necessarily been verified and validated for a specific test: 2-chloroethyl ethyl sulfide (CEES, CAS® Number 693-07-2), dimethyl adipate (DMA, CAS® Number 627-93-0), 2-isobutyl-3-methoxypyrazine (IMP, CAS® Number 24683-00-9), dimethyl methylphosphonate (DMMP, CAS® Number 756-79-6), methyl salicylate (MeS, CAS® Number 119-36-8), acetic acid (AA, CAS® Number 64-19-7), and ethylene glycol (CAS® Number 107-21-1).

APPENDIX A. TEST FIXTURES.

FIGURE LIST

APPENDIX A. TEST FIXTURES.

A.1. Test Fixture.

a. The test fixture is the entire test system that permits the test center to conduct APC testing. Figure A.1 shows the system block diagram of a generic APC test fixture. Major components include a temperature controlled APC enclosure, an environmental control system, a vapor or gas generator, a dissemination control system, challenge and effluent concentration monitors, and a discharge and capture system. The fixture is installed in a chamber or a laboratory fume hood. The fixture configuration affects the choice of methodologies for dissemination and detection, and implementation of safety and security SOPs and treaty considerations.

(1) The test fixture must be verified and validated. Changes to the test fixture must be documented through configuration management (Paragraph 7.1). After changes are made, the new configuration of the test fixture must be validated. Significant changes to the test fixture configuration will require both V&V.

(2) The area and length of the plumbing lines should be minimized to reduce fixture off-gassing, but large enough in diameter to avoid creating back pressure. The fixture materials must not absorb, react with, or later release the test chemical (e.g., passivated stainless steel and Teflon® may be used for agent testing) to the extent practical. Challenge concentration should be monitored in near real time as close to the test item as practical.

(3) The dissemination control system consists of a mixing chamber or manifold and dissemination lines.

(a) The mixing chamber or manifold mixes the challenge vapor or gas with clean air to maintain the desired challenge concentration and environmental conditions.

(b) The dissemination lines deliver the challenge to each APC. The dissemination system must have a bypass mode to divert the challenge flow to a sacrificial filter and send clean, dry air at the trial temperature to the APC while the challenge conditions are being established. When conditions are established, the challenge airflow is sent to the APC.

b. The test cell/test filter housing interface is required to connect the APC under test to the vapor generation/delivery system. The interface is unique for each filtration APC interface; construction depends on test requirements. Required specifications for the interface are dependent on test system description and configuration, connections, etc.

c. The mass of liquid chemical disseminated is measured by weighing the disseminator before and after the test. The mass of chemical disseminated may also be calculated by multiplying the volume of chemical consumed by the liquid density. The density should be determined at laboratory temperature based on the literature.

APPENDIX A. TEST FIXTURES.

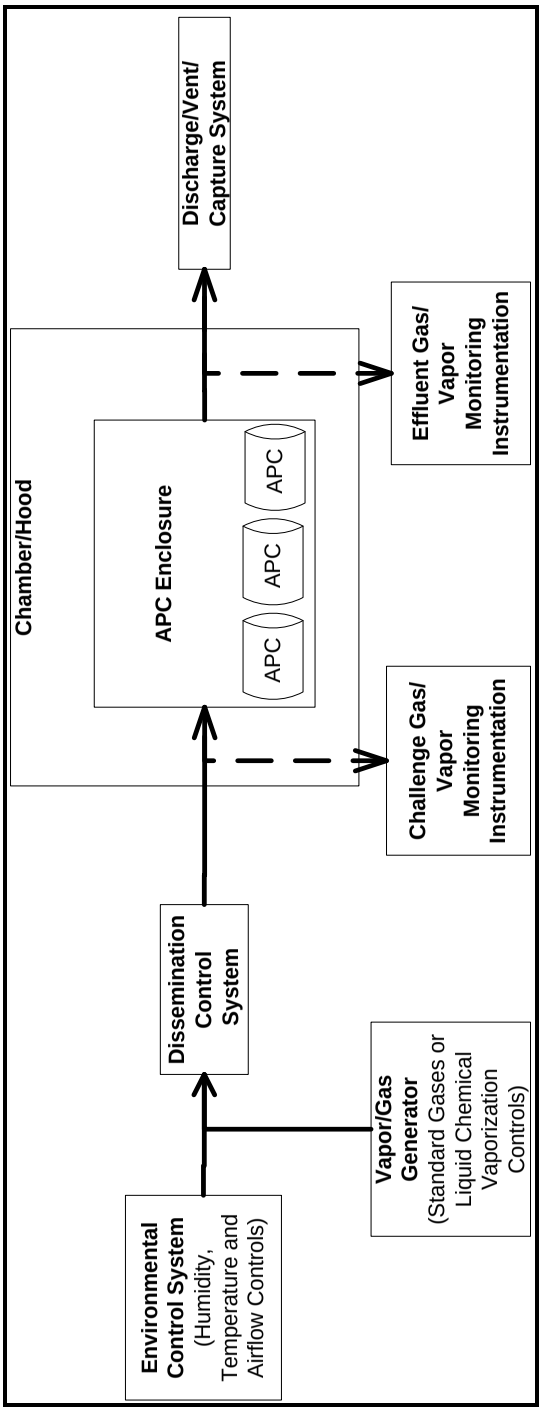


Figure A.1. Block diagram of generic air purification component (APC) test fixture with vapor containment engineering control enclosure (temperature/humidity control system).

APPENDIX A. TEST FIXTURES.

d. The test chemical can be disseminated using a vapor/gas generator that has been validated to meet test requirements. The stream is mixed with air from the environmental control system and fed to the APC. Representative dissemination methods depend on the characteristics of the chemical and the required concentrations. Some methods are described in the following paragraphs:

(1) Syringe-Pump Injection Combined With a Dilution System. Liquid chemical material is directly infused into a heated stream of air at a fixed infusion rate and the generated vapor from this stream is then mixed with an appropriate amount of dilution airflow to generate low-level vapor concentrations.

NOTE: For best performance, all moving parts or components of the syringe-pump should be cleaned and lubricated IAW manufacturer's instructions or as recommended by the laboratory procedures.

(2) Headspace Vapor Generation. Carrier gas flows across the headspace of a liquid in a container. The headspace must be large enough to supply a 3-minute or greater residence time to the APC. For single compounds with known vapor pressures, the concentration of the challenge may be controlled by adjusting the temperature of the headspace vapor generator.

(3) Diffusion Apparatus. A diffusion or permeation tube apparatus may be used to generate low vapor concentrations.

(4) Sparger. A sparger (a liquid reservoir constructed from a stainless steel cylinder) can generate continuous high-flow vapor streams. The sparger is loaded with 5-mm diameter soda lime glass beads (Sigma-Aldrich, St. Louis, Missouri), which promote mixing and prevent droplets of liquid from exiting the sparger. Two stainless steel mesh screens trap remaining liquid droplets and promote vapor mixing. Fixed fan blades further mix vapor. Spargers are equipped with a continuously replenishing loading port and are enclosed in a water jacket to control the temperature to meet target vapor concentrations. A thermocouple is placed inside the sparger to measure the liquid temperature. Another thermocouple measures the temperature of the jacket. If the temperature of the saturated carrier gas leaving the sparger is above or below ambient, the line leaving the mixing section must be thermally insulated. If the temperature of the saturated carrier gas leaving the sparger is above ambient, the line leaving the mixing section must be heat-traced. The airflow exiting the sparger is mixed with humidified main process air and fed to the filtration test apparatus. Temperature should be controlled using proportional-integral-derivative (PID) control.

(5) A saturator cell is a glass reservoir that can house liquid chemical and generate a vapor challenge using a carrier gas, such as nitrogen. A saturator cell consists of a cylindrical glass tube with an inlet and outlet tube connected at each end. The main body of a saturator cell contains a hollow ceramic cylinder, which promotes contact between the liquid chemical and carrier gas. The carrier gas enters the inlet tube and makes multiple passes across the ceramic cylinder surface that is wetted with the chemical used. The chemical/carrier gas vapor mixture then exits through the outlet tube to generate a chemical challenge. A water bath is used to

maintain a constant temperature needed to generate the chemical vapor challenge. Figure A.2 depicts a typical glass saturator cell.

(6) Pressurized Gas Dissemination. A number of chemical materials, particularly the TICs, are available commercially as mixtures in air from cylinders containing pressurized gas. A pressure regulator is required to disseminate the desired delivery pressure. The dilution of the gas and the required uncertainty must be specified to the vendor. If ordering gas from a vendor in units of ppm or ppb, clarify whether volume/volume (v/v) or weight/weight (w/w) mixing ratio is required. A MFC will be used to control the chemical flow rate to mix with the air stream.

e. An environmental control system is required to condition the vapor challenge to the target temperature, humidity, and airflow.

f. APC enclosures and test cells contain vapor to allow safe testing.

g. Temperature and humidity must be measured using calibrated instrumentation and the vapor generation/removal system must be verified to replicate the required environmental conditions. Absolute WVC may be calculated when the RH is used as a test requirement because RH varies with temperature and pressure (Appendix C).

h. Humidity can be generated from a variety of different systems such as Nafion® tubes (Perma Pure LLC, Toms River, New Jersey), saturator cells, water-filled spargers (Paragraph 4.4.2.b), or a HCS-501 humidity control system (Miller-Nelson, Livermore, California).

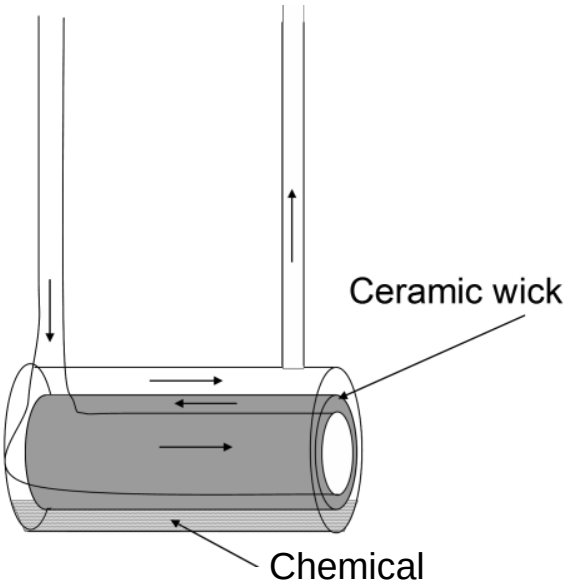
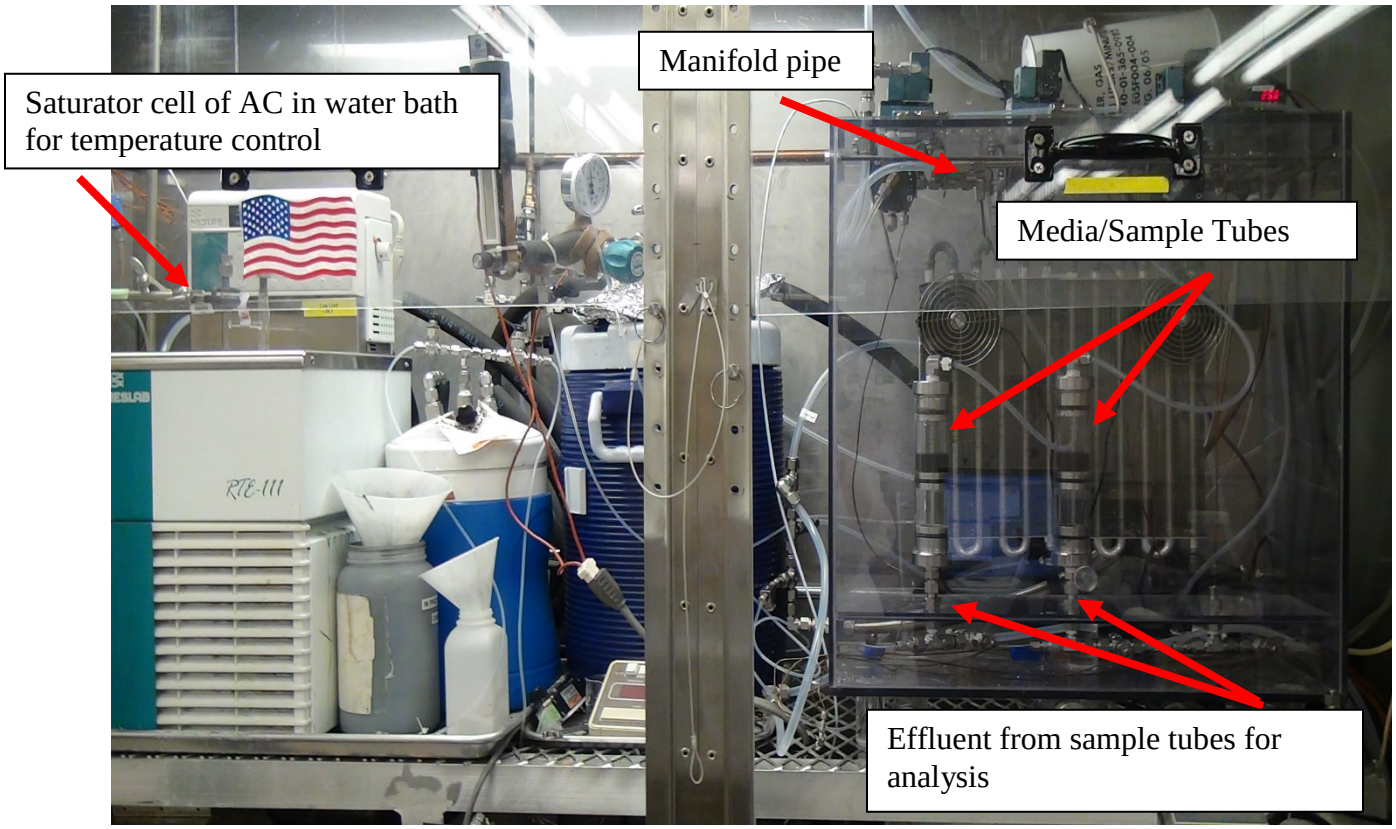


Figure A.2. Saturator Cell.

APPENDIX A. TEST FIXTURES.

A.2. FILTRATION MEDIA TEST FIXTURE DESIGN REQUIREMENTS.

a. A detailed description and tolerance requirements for the filtration media test fixtures can be found in Paragraph 2. The filtration media fixture pictured in Figure A.3 is a representative test fixture configuration[2].



NOTE: AC – hydrogen cyanide.

Figure A.3. Representative tube media test fixture (push-pull, vented system).

b. The representative dissemination methods listed in Paragraph A.1.d can be used to generate a vapor challenge. MFCs should be used to reduce the impact of upstream pressure changes on the air and contaminant generation systems, and generally provide superior performance.

c. Commercial process air/humidity control systems are used for the clean-air/water sparger system. The challenge air stream is then delivered to the media test cells. Contaminant-laden gas is pushed into the media test cells, and in some cases then pulled out using the

downstream blower. If necessary, pressure downstream from the media test cell is elevated using a standpipe discharging into a water tank, which provides the backpressure to direct the effluent to the analytical instrument. The backpressure from the standpipe is used to overcome any backpressure from the APC in a push system. A back-flow-prevention mechanism may need to be considered.

(1) A multiple test push-pull, vented apparatus, such as the one shown in Figure A.2, controls the flow through each test cell with a downstream blower and MFCs or valve/mass flow meter combinations for each test cell. Multiple identical tests can be efficiently obtained by installing a single tap off the test air reservoir so that all replicates can be initiated at one time by opening a single valve.

(2) The single test push apparatus is a simplified multiple test push-pull, vented apparatus, from which test media cells 2 and 3 (shown in Figure A.3) with their associated MFCs have been removed. The test procedure for the single test push apparatus may be identical to that for the multiple test push-pull, vented apparatus. An analysis of the apparatus and process should be conducted.

d. Filtration Media Test Cell

(1) A filtration media test cell is shown in Figures A.4 and A.5. Each cell must be constructed of inert materials, including gaskets and O-rings and be adequately sealed to prevent bypass.

(2) The upstream face of the test bed should be a minimum of 5 test-cell diameters away from challenge gas entry and should include a floater with a mesh diameter that is small enough to retain the media, but large enough so as not to restrict airflow, i.e., negligible pressure drop effects.

(3) The media test cell must be large enough to reduce edge or wall effects to a negligible level. The test cell size should be IAW the sizing required in ASTM D2854-96[27].

e. Storm Filler.

(1) The storm filler apparatus is used to fill filtration media test cells. The storm filler apparatus includes a material reservoir, a feeder device designed to fill the media bed, and the storm filler. The feeder may be vibrated to settle the media.

(2) As shown in Figure A.6, the storm filler uses sequential screens to distribute the media fed into the filtration media test cell. The storm filler is constructed of an inert material resistant to erosion by filtration media. The following requirements must be met to fill the filtration media test cell.

APPENDIX A. TEST FIXTURES.

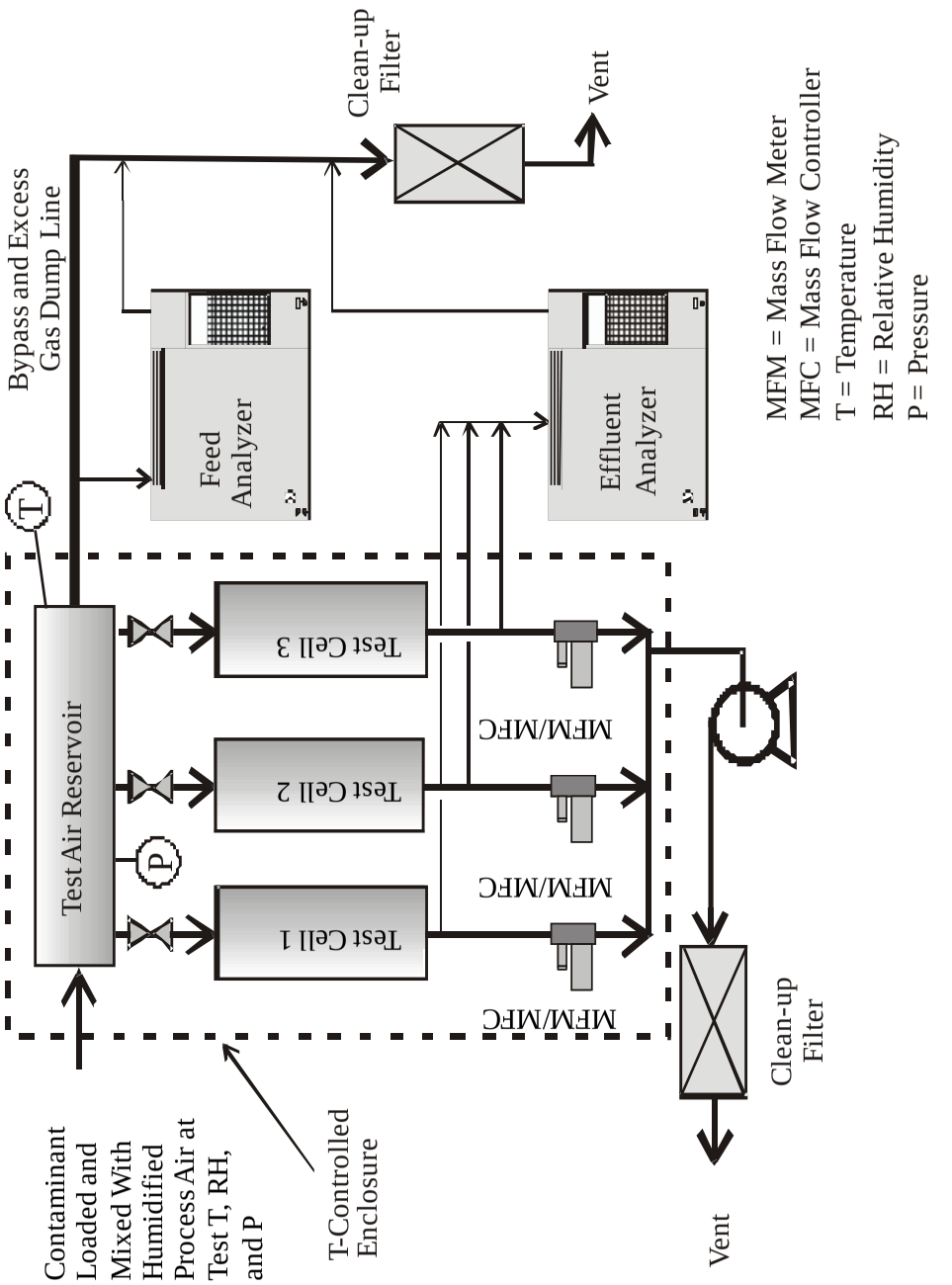


Figure A.4. Filtration media test fixture.

APPENDIX A. TEST FIXTURES.

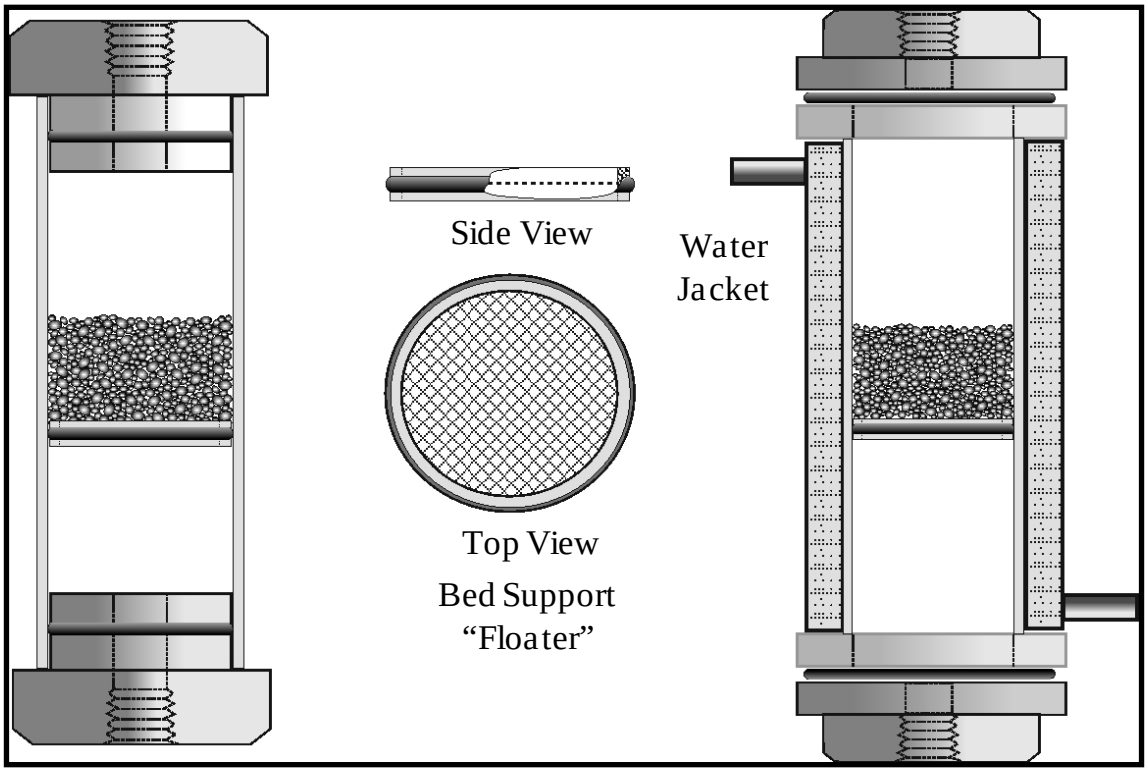


Figure A.5. Filtration media test cell.

APPENDIX A. TEST FIXTURES.

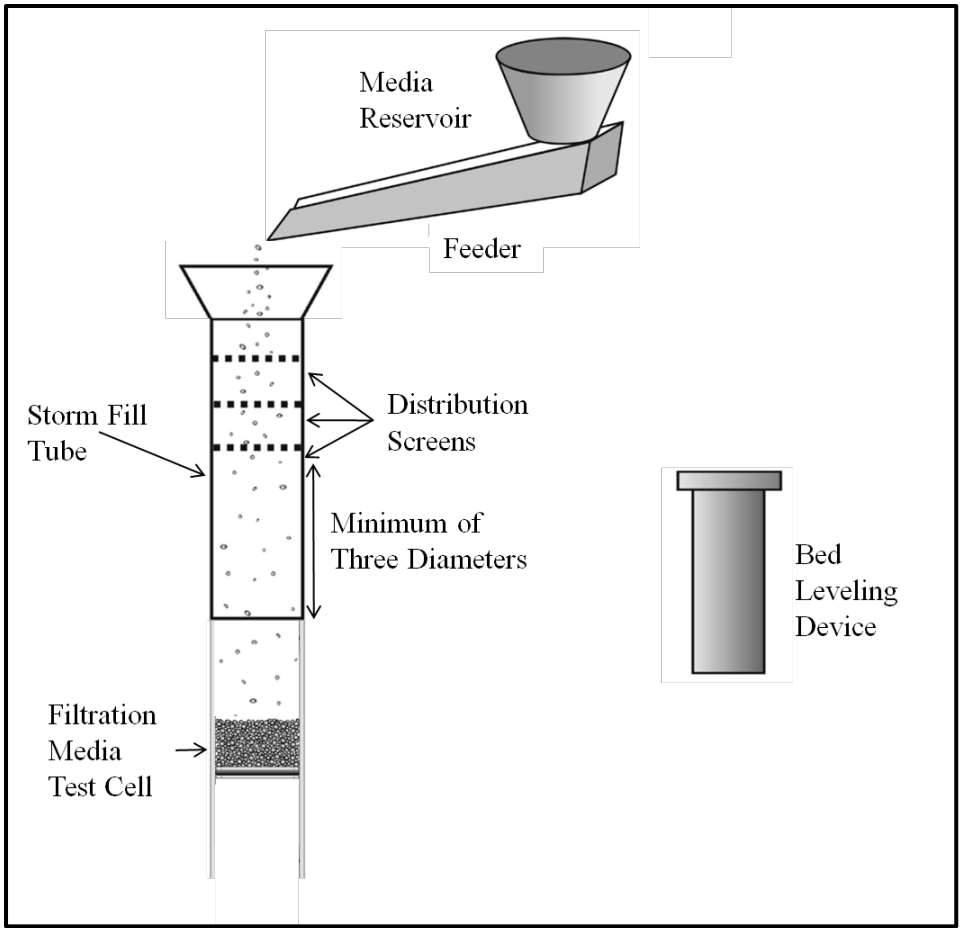


Figure A.6. Schematic drawing of storm filler and bed leveling device.

(a) The inside diameter of the storm filler tube should be the same as the inside diameter of the filtration media test cell being filled.

(b) Three parallel distribution screens must be spaced a minimum of 4 cm apart. Each screen mesh is rotated 45 degrees from the previous screen to prevent any particle from having an unobstructed fall path.

(c) The screen openings should be three to five times the largest particle diameter of the filtration media being filled. For example, 12x30-mesh granules should be passed through distribution screens whose mesh spacing is between 5.1 and 8.5 mm.

(d) The distance from the bottom screen to the bottom of the storm filler tube should be three times the tube diameter.

APPENDIX A. TEST FIXTURES.

A.3. IP CANISTER AND FF TEST FIXTURE.

a. The SWIFT system at DPG is an NRTM fixture that includes all the components needed to execute FF or IP canister testing under a wide range of environmental and threat challenge conditions. The SWIFT fixture is compatible with a range of dissemination configurations and instrumentation systems and has been tested using a variety of materials.

b. The Dugway fixture (^{d}FIX) is an integral part of the SWIFT fixture. The ^{d}FIX is a thermostat-controlled, plastic enclosure that contains the APCs. The ^{d}FIX allows the control and measurement of conditions such as temperature, humidity, and challenge vapor concentrations and fits inside most chemical fume hoods. The ^{d}FIX can test several APCs to be challenged by simulants or agents in either vapor or liquid states.

c. Temperature is measured by thermocouples. Temperature is controlled over the range 5° to 55°C by a thermoelectric heating/cooling unit. A Nafion® tube is fed with DI water and plumbed into the airstream to add humidity to the test fixture. Humidity is monitored using humidity probes. RH may be controlled over the range from 0 to approximately 85 percent by adjusting temperature and the flow rate through the Nafion® tube. A mixing chamber or ballast volume mixes the test chemical, air, and water vapor.

d. For total airflow less than approximately 10 aLpm, a syringe pump infuses liquid test compound into the airstream. For total airflow above 10 aLpm, a sparger-type disseminator may be used. Concentrations may range from 1 to 5,000 mg/am³ as specified in the test plan. Challenge concentration is measured by either an FTIR gas analyzer or a GC analyzer equipped with an LVS.

e. The inlet of each APC is at ambient pressure because of the push-pull design of the SWIFT fixture. Airflow is controlled independently using an MFC. A GC (e.g., MINICAMS®) is used to monitor downstream effluent concentration.

f. The SWIFT fixture includes safety processes, software control, and hardware to minimize risk to the operator. For example, agent vapor is contained in case of power failure and the exhaust of each detector is vented to sacrificial filter.

g. The SWIFT fixture configuration for testing IP canisters is shown in Figure A.7[25]. Three small canisters are mounted within the challenge housing, which is designed to hold IP canisters and draw challenge vapor through them. The challenge housing resides in the ^{d}FIX.

h. The FF configuration of the SWIFT fixture is shown in Figure A.8[24]. Three FF swatches are mounted in military-standard aerosol vapor liquid assessment group (AVLAG) swatch cells. The swatch cells are plumbed into the challenge housing.

APPENDIX A. TEST FIXTURES.

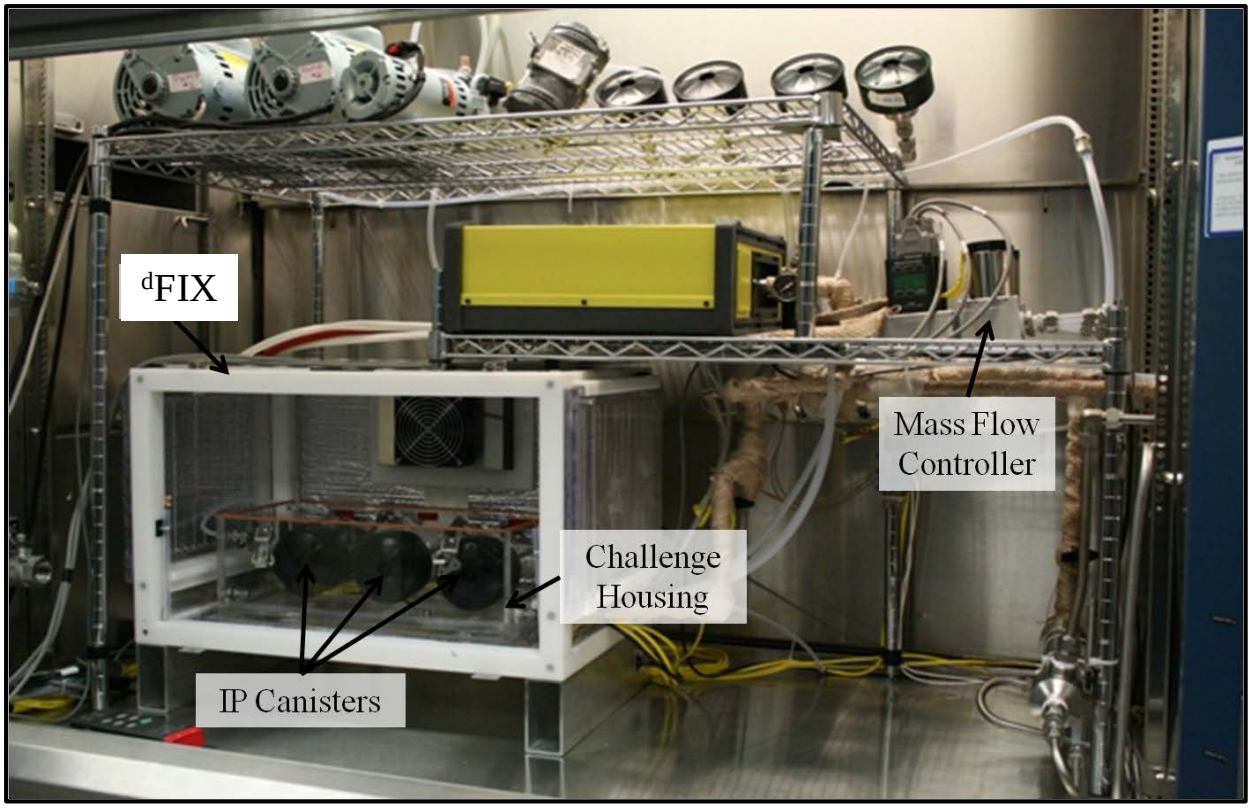


Figure A.7. Swatch including filter test (SWIFT) fixture configured for individual protection swatch testing.

APPENDIX A. TEST FIXTURES.

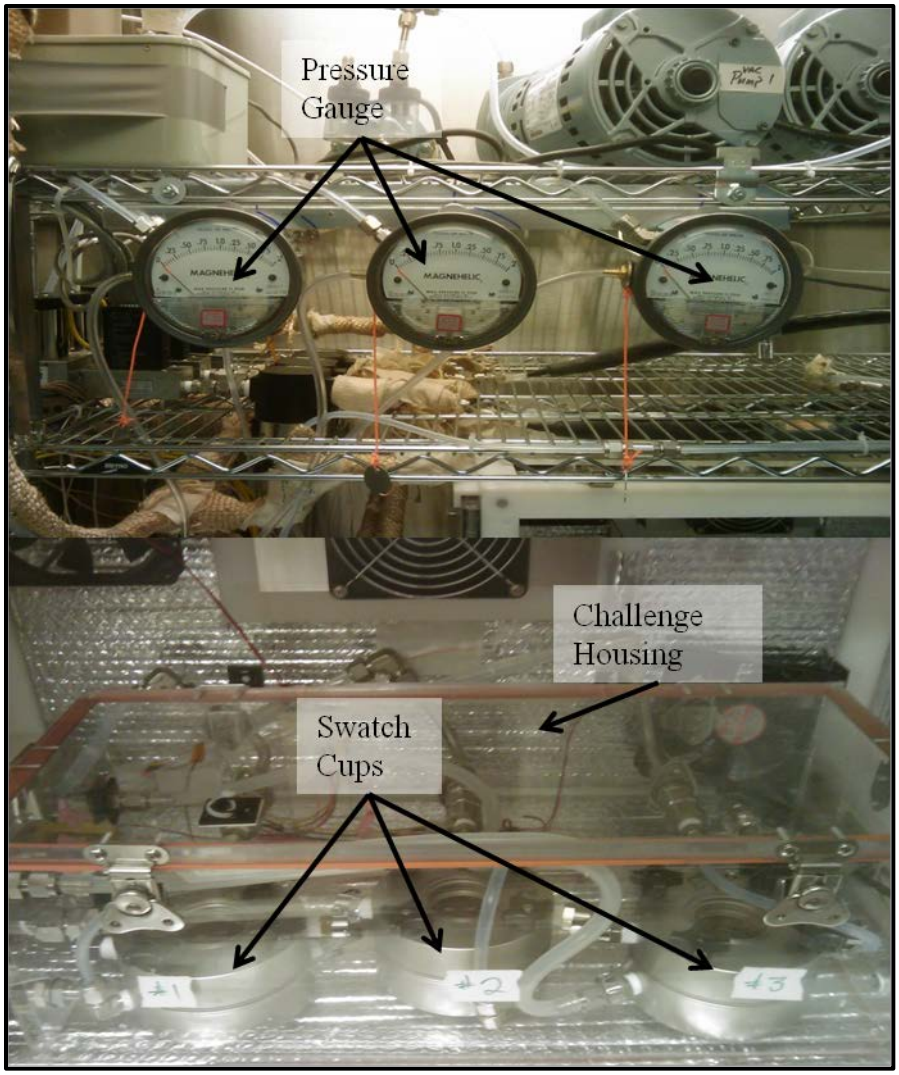


Figure A.8. Swatch including filter test (SWIFT) fixture configured for filtration fabric testing.

APPENDIX A. TEST FIXTURES.

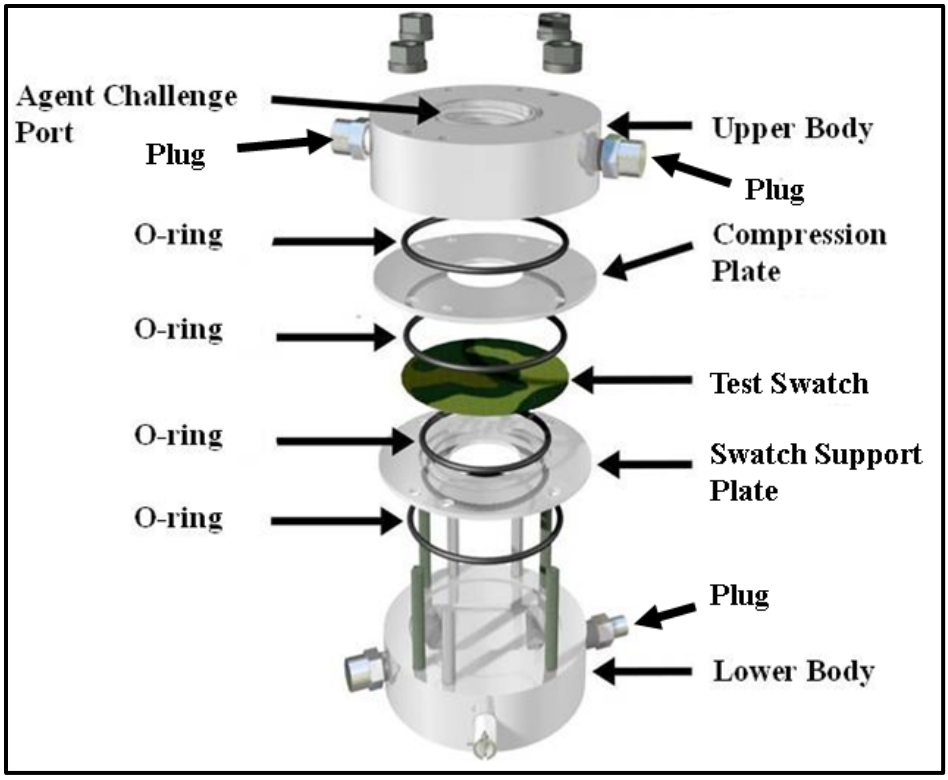
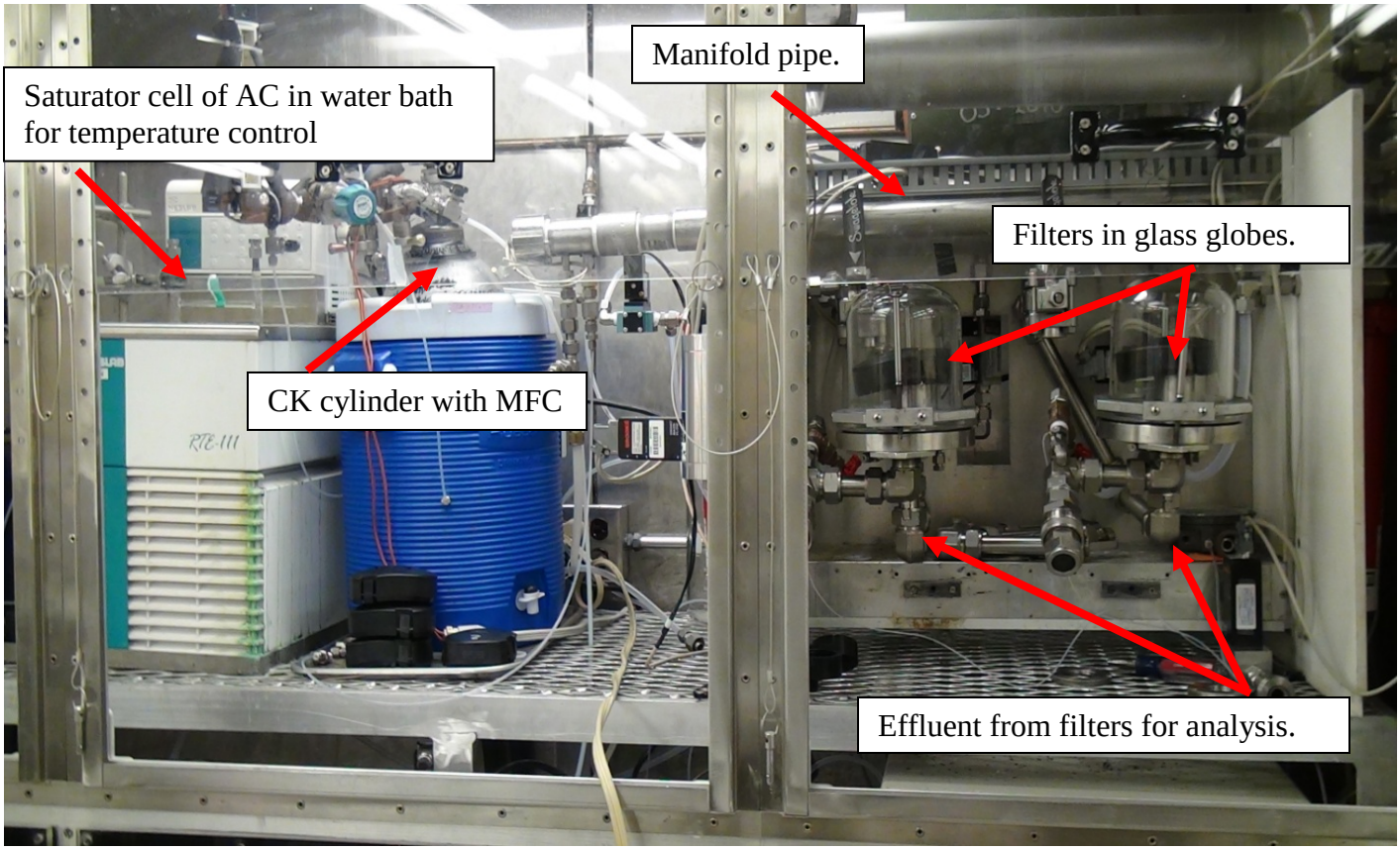


Figure A.9. Diagram of a flow-through filtration swatch mounted in a swatch cup.

i. For IP canister testing, a similar setup as shown in Figure A.10 could be used. In place of test cells, filters are mounted on faceplates that have an inlet and outlet port, and covered with glass or Lexan globes to contain the chemical challenge. Figure A.10 shows a picture of an IP canister test system at ECBC.

APPENDIX A. TEST FIXTURES.



NOTE: AC – hydrogen cyanide, CK – cyanogen chloride, MFC – mass flow controller.

Figure A.10. A picture of an IP filter canister test system at Edgewood Chemical and Biological Center.

A.4. CP APC TEST FIXTURE.

a. The AAPTF at DPG is a modular design constructed to test full-scale CP APCs against vapor-phase agents, simulants, and TICs under controlled environmental conditions (Figure A.11). The fixture consists of several modular components that deliver the conditioned chemical to the filter housing through 20.3-cm diameter ducting. Butterfly valves, baffles, and chemical mixing devices are incorporated to ensure the CP APC will be challenged.

b. The AAPTF operates within the fully enclosed chemical agent super chamber (CASC). The CASC consists of an environmental test chamber (ETC) and an airlock. The AAPTF constructed within the ETC permits environmental controls. Temperatures can be controlled within the range from 5° to 49°C. RH can be controlled from 5 to 40 percent RH[3].

APPENDIX A. TEST FIXTURES.

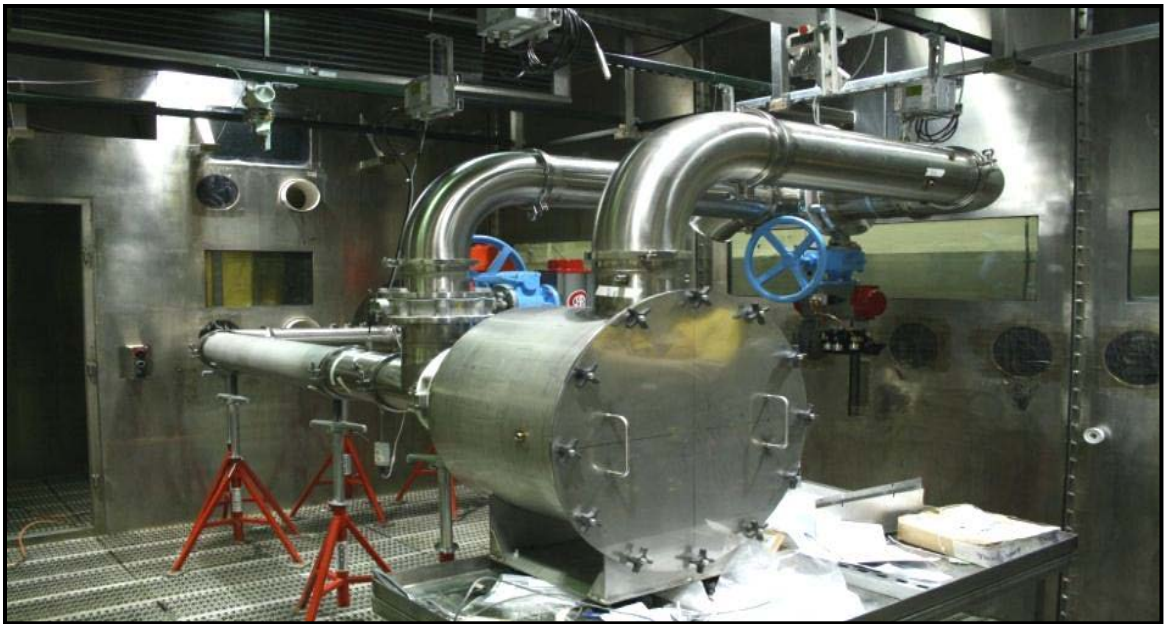


Figure A.11. Advanced Air Purification Test Fixture (AAPTF) at US Army Dugway Proving Ground (DPG).

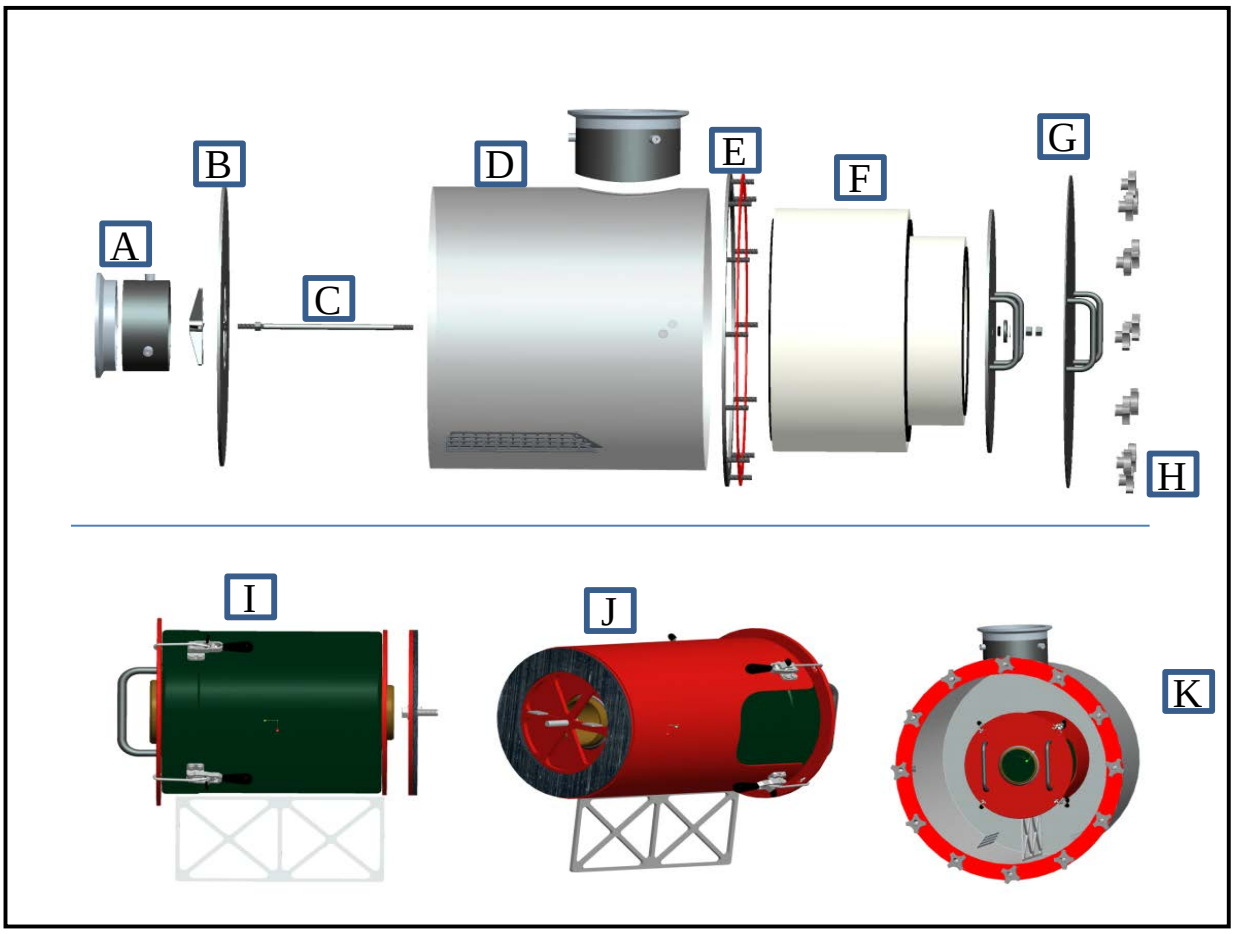
c. The chemical dissemination control system and vapor/gas generator are integrated into the AAPTF to deliver chemical to the dissemination ports through dual contained lines. The disseminated chemical is then diluted with conditioned air at a fixed airflow and passed through the static mixer providing a homogeneous chemical challenge. Spargers are available to generate continuous high-flow chemical streams from liquid challenge compounds.

d. Referee instrumentation monitors the challenge and effluent chemical vapor concentration. FTIR gas analyzers are used to measure the challenge chemical concentration through three sampling ports. A MINICAMS® or other GC is used to measure the effluent chemical concentration.

e. Chemical vapor challenging, environmental control, and monitoring are executed remotely. LabVIEW® software remotely operates all test fixture functions such as monitoring
R Real-time data from each instrument system are displayed in LabVIEW®.

f. Filter housings are designed to accommodate M98/M48A1 filters and panels of FF. Filter adapters can be interfaced with the filter housing (Figure A.12).

APPENDIX A. TEST FIXTURES.



Pipe Adapter

Alignment Rod

3/4 Side View

Front View

Figure A.12. Exploded view of the Advanced Air Purification Test Fixture (AAPTF) filter housing with M98 filter (top) and M48A1 filter (bottom).

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APPENDIX B. APPLICATION OF KEY BFCs.

FIGURE LIST

FIGURE PAGE

APPENDIX B. APPLICATION OF KEY BFCs.

B.1. SCOPE.

a. BFCs are airborne contaminants (in both particulate and vapor form) associated with any environment where the filter may be placed into service. This includes not only the actual battlefield, but also motor pools, maintenance areas, de-icing stations, training sites, and decontaminations areas.

b. BFCs for testing should be chosen through a systematic selection process in which operational users and technical subject matter experts (SMEs) are consulted[37]. Criteria for choosing BFCs should be proposed, reviewed, and prioritized.

c. Methodologies to precondition APCs with BFCs may need to be developed for test execution. BFC is usually applied in a separate fixture designed to draw BFCs through the APC. However, applying BFC in the same fixture used for APC testing may be performed.

d. The breakthrough of agent, simulant, or TIC challenge will be tested after the APC has been exposed to the BFC. BFC-exposed and unexposed item breakthrough times will be compared.

B.2. FACILITIES AND INSTRUMENTATION.

The requirements for the BFC exposure chamber are in Paragraph 2.1. Instrumentation to measure concentration, RH, and temperature will be determined by the test requirements and should meet the requirements in Paragraph 2.2.

B.3. HEALTH AND SAFETY.

Many BFCs pose a significant health risk to personnel. The operator must wear appropriate PPE. The operator must read and understand the SDSs associated with the BFCs and the materials that compose the filter bed. Some BFCs are toxic, flammable, and/or reactive chemicals. The safety issues of simulant and agent testing also apply to BFC exposure (Paragraph 3.3).

B.4. BFC SELECTION.

a. Test requirements should provide a list of BFCs and the expected airborne concentration representative of a specific operational environment. If such information is not provided, it should be developed jointly by the testing and evaluation community and the intended APC user. Failure to correctly analyze the environment can lead to incorrect interpretations of data.

b. If the list of BFCs provided is large, it may be impractical to test all combinations of BFCs and challenge chemicals. Therefore, BFCs should be prioritized based on the nature of the BFC, its prevalence on the battlefield, and potential interactions with the filtration media. If possible, a single design-limiting BFC should be identified. A methodology for identifying design-limiting BFCs is outlined in TOP 08-2-196[9].

APPENDIX B. APPLICATION OF KEY BFCs.

B.5. BFC EXPOSURE.

a. In battlefield exposure, the concentration of a BFC exposure will be less than 100 parts per million (ppm) over a long duration (e.g., 1 year or longer). Accelerated exposure is the practical alternative where the BFC is delivered to the APC at a higher concentration over a shorter period but achieves an equivalent Ct. Accelerated exposure can reduce the exposure duration to a few days.

b. The APC will be weighed and mounted in the BFC exposure chamber. APCs will be mounted in the BFC exposure chamber (Figure B.1) and attached to a blower or flow controller to pull the BFC through the filter. The challenge environmental temperature and humidity will be recorded. APCs will be mounted as follows:

(1) FF swatches and panels will be hung on a fixture with a controlled flow through the fixture.

(2) IP canisters will be exposed with a controlled flow through each.

(3) CP canisters will be pretreated either through a fan filter assembly (FFA) or a modified filter housing and blower dedicated to treatment with BFC.

c. The temperature and RH of the fixture should be adjusted to the target if temperature and RH are controlled.

d. Airflow through the APC should be initiated at the target flow rate. Then the BFC dissemination can begin. BFC can be disseminated through a variety of methods. For some methods, the BFC generator will be connected to the BFC exposure chamber through hoses or ducts. For others, the BFC generator may be located within the BFC exposure chamber.

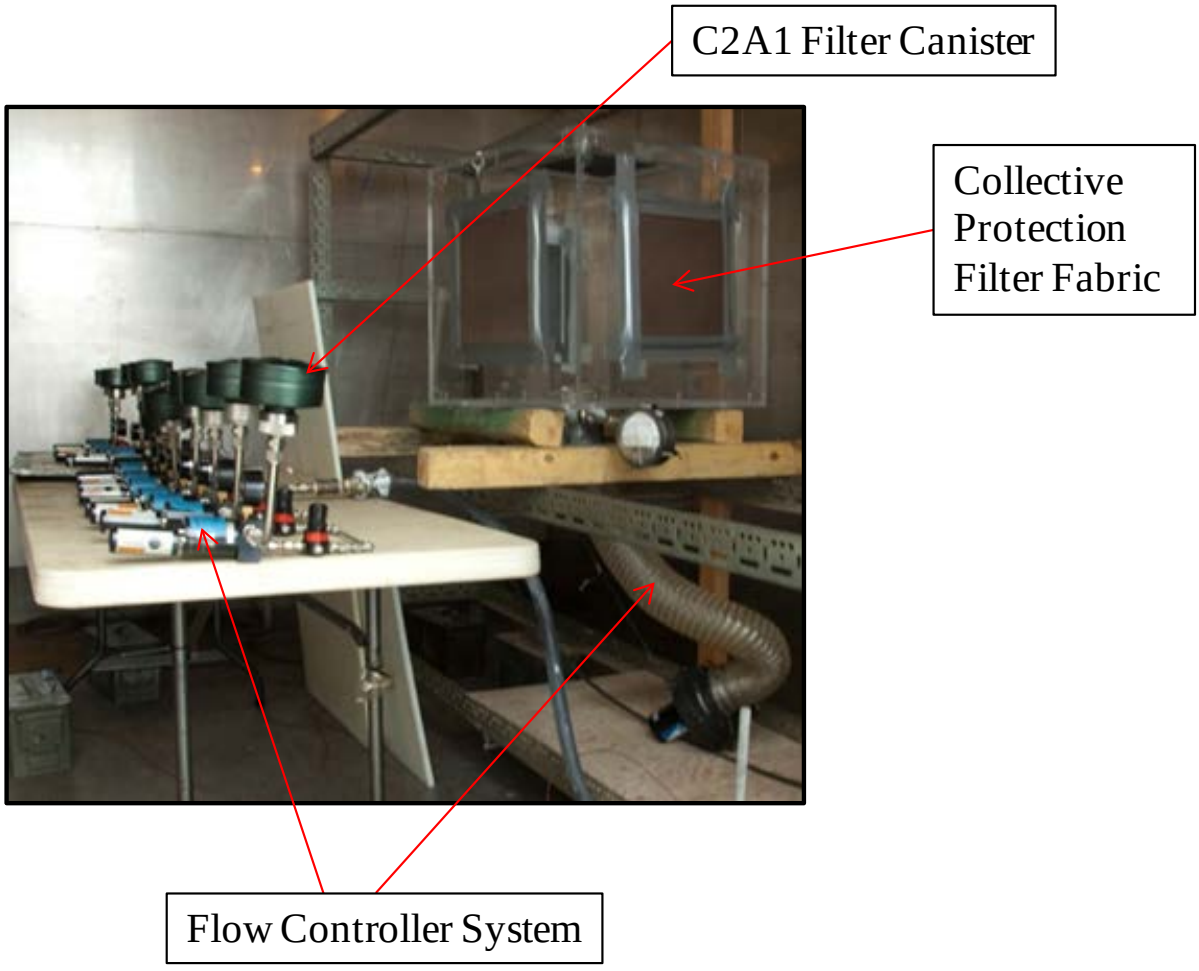
e. During the BFC exposure, the chamber will be inspected for signs of condensation and record observations on the data sheet. Based on the nature of the BFC, the presence of condensation may require a restart.

f. BFC dissemination and airflow should be terminated when the exposure requirements for the test have been met. If the test requires repeated or intermittent BFC exposure, it will be necessary to purge the filter with air at the target RH and temperature between BFC exposures.

g. The details of these exposure methodologies may need investigation before test execution. Methodologies for JP-8 exhaust and fog oil mist[38] exposure of APCs are described in the Final Test Report (FTR) for the Joint Expeditionary Collective Protection (JECP) Air-Purification System (APS) Testing of the Passive Air-Filtration System and the FTR for the JECP APS Testing of the Active Air-Filtration System[24,25]. Methodologies are also included in ECBC technical reports for testing the effects of BFC exposure on CP filters, IP filters, and media[39,40,41].

APPENDIX B. APPLICATION OF KEY BFCs.

h. After exposure, the APCs will be bagged, sealed, and weighed, and then placed in storage until testing.



NOTE: The figure depicts a chamber flooded with the BFC, jet propulsion, type 8 (JP-8) exhaust.

Figure B.13. Interior of battlefield contaminant (BFC) exposure chamber showing collective protection filter fabric (FF) panel and individual protection (IP) filter canister.

APPENDIX B. APPLICATION OF KEY BFCs.

B.6. QUALITY ASSURANCE.

In planning for accelerated exposures, plans should be made to test reference media in parallel. With this approach, reference media should be exposed to a flow of humid air (without BFC) for a duration consistent with that of the BFC-exposed media. Following this, the reference media should be subjected to breakthrough testing with the target chemicals. Results obtained using the BFC-exposed media are to be compared with results obtained using the reference media.

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APPENDIX C. HUMIDITY MEASUREMENT.

C.1. METHODS OF MEASURING HUMIDITY.

Different methods may be used to determine the humidity of the process stream just above the filter bed. The first method involves the use of a certified RH probe located at a point downstream from the humidity disseminator and before the chemical injection point. The second method determines the humidity based upon the change in mass of the humidity disseminator. The third and preferred method is direct concentration measurement after chemical vapor has been added. Each method is described below. In all methods, control and measurement of temperature is important.

a. A certified RH probe may be used to determine the RH of the chemical-free air stream just above the filter bed. When using this technique, the temperature and process pressure (in torr gauge) are recorded at the filter bed challenging stream, and the barometric pressure is recorded. The RH of chemical-free air may be measured. This is because the presence of chemical in the process stream may affect the measured RH. The RH is an indication of the moisture content of the air. In order to determine the moisture content (mole fraction of water) in the air at the point of RH measurement, the Antoine equation for water is employed (Equation C.1).

$$\ln(p_{w,s}) = 18.3036 - \frac{3816.44}{T - 46.13} \qquad \text{Equation C.1}$$

Where,

$p_{w,s}$ = the saturation partial pressure of water (Torr)

T = the temperature (Kelvin).

ln = natural logarithm

(1) Dividing $p_{w,s}$ by the total pressure will yield the mole fraction of water at saturation ($x_{w,s}$, Equation C.2).

$$x_{w,s} = \frac{p_{w,s}}{p_{tot}} \qquad \text{Equation C.2}$$

Where,

$x_{w,s}$ = the mole fraction of water at saturation

$p_{w,s}$ = the saturation vapor pressure

p_{tot} = the total pressure

APPENDIX C. HUMIDITY MEASUREMENT.

(2) Multiplying this value by the RH will yield the mole fraction of water in the air stream at the point of RH measurement ($x_{w,s}$). For example, if the RH measurement recorded between the water saturator and the point of chemical injection is 80 percent, the temperature at this point is 25°C, the pressure is 8 torr gauge, and the room pressure is 730 torr, the mole fraction of water in the humidity air stream at this point is calculated in Equation C.3.

$$\ln(p_{w,s}) = 18.3036 - \frac{3816.44}{298.15 - 46.13} = 3.16$$

$$p_{w,s} = 23.57$$
$$x_{w,s} = 23.57 \div (730 + 8) = 0.03194$$
$$x_w = 0.03194(80\%) = 0.02555$$

Equation C.3

Where,

$p_{w,s}$ = the saturation vapor pressure
$x_{w,s}$ = the mole fraction of water at saturation vapor pressure
x_w = the mole fraction of water
ln = natural logarithm

(3) The humid air stream is diluted by the flow of air that contains chemical. The mole fraction of water must be reduced accordingly. For example, if there is 15.0 aLpm of humid air and 5.0 aLpm of air containing chemical the mole fraction of water is given by Equation C.4.

$$x_w = 0.02555 \times 15 \div (15 + 5) = 0.019163$$

Equation C.4

Where,

x_w = the mole fraction of water

(4) The RH at the entrance of the filter bed is determined by first calculating the saturation mole fraction of water at the temperature and pressure of the filter bed, using Equations C.1 and C.2.

(5) Note the difference between the RH at the point of measurement and the RH determined just before the filter bed. For the test system shown in Figure A.2, the temperature just before the test cells is controlled, while the temperature at the point of RH measurement is

not controlled. Thus, should the ambient temperature differ from that of the filter bed, the RH may not be within the stated specification limits.

b. The second method for determining the RH of the chemical-free process stream entering the filter bed is to employ the weight loss in the water sparger over the duration of the test. The water sparger is temperature controlled and will therefore deliver a stable concentration of water to the process.

(1) However, it does not take into account the condensation and loss of water in the fixture, and therefore can only give an upper bound to the humidity delivered to the test item. For this method, the mole fraction of water in the process stream is determined from calculating the moles of water (Equation C.5) and the moles of air (Equation C.6) that entered the process stream. The mole fraction is the moles of water divided by the total number of moles (Equation C.7). Dividing the mole fraction obtained in Equation C.7 by the saturation mole fraction of water calculated using the temperature and pressure of the filter bed inlet would yield the RH of the chemical-free process stream just before the filter bed.

$$n_w = \frac{m_w}{18.015}$$ Equation C.5

Where,

n_w = the moles of water

m_w = mass of the water lost to evaporation in the sparger

$$n_a = \frac{tr}{0.08206T}$$ Equation C.6

Where,

n_a = the moles of air

t = the duration of the test in minutes

r = the rate of airflow in NLpm

T = the absolute temperature in Kelvin

APPENDIX C. HUMIDITY MEASUREMENT.

$$x_w = \frac{n_w}{n_w + n_a}$$ Equation C.7

Where,

x_w = the mole fraction of water
n_w = the moles of water
n_a = the moles of air

(2) For example, if 7.60 NLpm of airflow passed through the sparger for 150 min, the temperature and pressure measured at the filter bed inlet were 24.8°C and 5 torr gauge, respectively, and the water lost from the sparger cell was 21.326 g, the humidity of the chemical-free process stream at the inlet of the filter bed would be calculated using Equations C.8 through C.10. The results of these calculations are shown in Equations C.8 through C.10.

$$n_w = \frac{21.326}{18.015} = 1.1848$$ Equation C.8

$$n_a = \frac{150 \times 7.60}{0.8206 \times 298.15} = 46.6$$ Equation C.9

$$x_w = \frac{1.1848}{1.1848 + 46.6} = 0.0248$$ Equation C.10

Where,

x_w = the average mole fraction of water in the process stream
n_w = the moles of water
n_a = the moles of air

APPENDIX C. HUMIDITY MEASUREMENT.

(3) Equation C.10 is the average mole fraction of water in the process stream. The saturation concentration of water in air at the temperature (24.8 °C) and pressure of the filter bed (735 torr) is calculated using the Antoine equation (Equation C.1). The saturation mole fraction of water ($x_{w,s}$) is determined to be 0.03169. Dividing the mole fraction of water by the saturation mole fraction of water yields the RH (Equation C.11).

$$Relative\ Humidity\ (RH) = 0.248 \div 0.03169 = 80.5\% \qquad \text{Equation C.11}$$

(4) The value calculated in Equation C.11 represents the average RH for the test. If it is desired to generate a plot of RH as a function of time, the RH may be computed whenever temperature and pressure data points are logged during the run.

c. The third and preferred method is direct measurement of humidity and temperature as close to the test item as practical. Vaisala probes have been used effectively to measure RH in the presence of GB, soman (GD), distilled mustard (HD), VX, TMP, TEP, TPP, MeS, and about 10 candidate simulants[35,36]. The Vaisala reports both the temperature and the RH, measured simultaneously by the same device at the same location.

NOTE: Certain chemicals may impair the ability of RH probes to measure humidity and corrosive chemicals may even damage it. In such cases, indirect methods may be required.

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APPENDIX D. GLOSSARY.

Background concentration	Residual vapor concentration arising from material left in the fixture during the previous trial
Breakthrough	Concentration of chemical in the effluent stream that exceeds the specified breakthrough concentration.
Breakthrough concentration	Upper bound allowed for the effluent concentration. Breakthrough concentration is often the 8-hour MEG level.
Breakthrough TWA	Upper bound allowed for the effluent exposure that is considered acceptable for the test item. Breakthrough exposure is often the 1-hour, 8-hour or 24-hour MEG.
Breakthrough time, actual	Actual time when the concentration of chemical in the process effluent stream exceeds the breakthrough concentration.
Breakthrough time, corrected	Breakthrough time corrected for chemical desorption. This is based on the amount of chemical irreversibly retained or removed (via reaction) by the filter bed.
Breakthrough time, reported	Time that the concentration of chemical in the process effluent stream exceeds the breakthrough concentration, corrected for the deviation in the challenge concentration.
Challenge	The dissemination of chemical agents or simulants during testing.
Dosage	The toxicological amount of a compound to which an individual may be exposed. The calculation of this value depends on the compound.
Effluent	The gas that has passed through the test item.
Effluent concentration	The concentration of the test chemical in the effluent
Equipment	A set of mechanical tools or clothing designed for a particular test purpose.
Exposure	The integral of concentration through the duration of a challenge. Exposure has units of concentration × time.
Filtration media test	A test of the media component of an APC.

APPENDIX D. GLOSSARY.

Fixture	A permanently fixed system including many components designed and constructed to execute APC testing.
Linear velocity	The velocity of air stream through the filter bed, computed as volumetric flow rate divided by the cross sectional area of the tube (also known as face velocity).
Near real time	One measurement every 1 to 15 minutes.
Offgas	Desorption of compound from a surface that can create a residual hazard.
Push test fixture	Test fixture with a single APC that does not need MFCs to ensure that airflow is distributed equally.
Push-pull test fixture	Text fixture that has an MFC for each APC to distribute airflow equally.
Real time	At least one measurement per minute.
Safety air monitoring	Monitoring of test chemical concentration in the laboratory to ensure the safety of personnel.
Storm filling	Process of filling filters by pouring media into the filter through a series of screens.
Single pass filtration	Filtration technology where stream of air to be purified only passes through the filter once.
Sparger	A liquid reservoir constructed from a stainless steel cylinder used to generate continuous high-flow vapor streams.
Test chemical	The compound with which the test item will be challenged.

APPENDIX E. ABBREVIATIONS.

ΔP	differential pressure
AA	acetic acid
AAPTF	Advanced Air Purification Test Fixture
AC	hydrogen cyanide
aL	actual liters
aLpm	aL per minute
am^3	actual cubic meters
AMCR	U.S. Army Materiel Command Regulation
ANSI NCSL	American National Standards Institute National Conference of Standards Laboratories
APC	air purification component
APHC	Army Public Health Center (Provisional)
APS	air purification system
AR	Army Regulation
ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing and Materials
ASZM-TEDA	activated carbon impregnated with copper, silver, zinc, molybdenum and triethylenediamine
ATEC	U.S. Army Test and Evaluation Command
ATTN	attention
AVLAG	aerosol vapor liquid assessment group
BFC	battlefield contaminant
CAPAT	capability area process action team
CAS®	Chemical Abstracts Service®
CASARM	chemical agent standard analytical reference material
CASC	chemical agent super chamber
CB	chemical and biological
CBRN	Chemical, biological, radiological, and nuclear
CCTF	Combined Chemical Test Facility
CD	compact disc
CDD	capability development document

APPENDIX E. ABBREVIATIONS.

CEES	2-chloroethyl ethyl sulfide
CK	cyanogen chloride
CoA	certificate of analysis
Conc	concentration
CP	collective protection
CRM	certified reference material
Ct	concentration × time
CWA	chemical warfare agent
CWC	chemical weapons convention
DA	Department of the Army
DAS	data acquisition system
^{d}FIX	Dugway fixture
DI	deionized
DMA	dimethyl adipate
DMMP	dimethyl methylphosphonate
DMP	data management plan
DOD	Department of Defense
DoE	design-of-experiment
DPG	U.S. Army Dugway Proving Ground
DQO	data quality objective
DTIC	Defense Technical Information Center
DUSA TE	Deputy Under Secretary of the Army, Test and Evaluation
DVD	digital video disc
ECBC	U.S. Army Edgewood Chemical Biological Center
EDP	event design plan
EMT	emergency medical technician
ETC	environmental test chamber
FF	filter fabric
FFA	fan filter assembly
FID	flame ionization detector
FO	fog oil

APPENDIX E. ABBREVIATIONS.

FPD	flame photometric detector
FTIR	Fourier-transform infrared
FTR	final test report
GB	sarin
GC	gas chromatograph
GD	soman
G-Series	nerve agent family
GUM	Guide to the Expression of Uncertainty in Measurement
HD	distilled mustard
HEPA	high-efficiency particulate air
HHA	health hazard assessment
H-series	blister agent family
HUC	human use committee
IAW	in accordance with
IEC	International Electrotechnical Commission
IHP	industrial hygiene plan
IMP	2-isobutyl-3-methoxypyrazine
IMS	ion mobility spectrometer
IP	individual protection
ISO	International Standards Organization
iwg	inches of water gauge
JECP	Joint Expeditionary Collective Protection
JP	JP-8
JP-8	jet propulsion fuel, type 8
JRO	Joint Requirements Office
kPa	kilopascal
L	Lewisite
LCL	lower control limit

APPENDIX E. ABBREVIATIONS.

LLC	limited liability company
LSL	lower specification limit
LVS	low volume sampling
MEG	military exposure guideline
MeS	methyl salicylate
MFC	mass flow controller
MQI	measurement quality indicator
N	none
NEPA	National Environmental Policy Act
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Standards and Technology
NLpm	normal liters per minute
NPD	nitrogen-phosphorus detector
NRTM	near RTM
NSN	national stock number
ODASAF	Office of the Director of the Army Safety
OM	operating manual
ORI	operational readiness inspection
OSHA	Occupational Safety and Health Administration
OTA	operational test agency
PAM	pamphlet
PDF	portable document format
PID	proportional integral derivative
POSS	preoperational safety survey
PPE	personal protective equipment
ppm	parts per million
QA	quality assurance
QC	quality control
R123	HCFC-123, 1,1-Dichloro-2,2,2-trifluoroethane

APPENDIX E. ABBREVIATIONS.

RDT&E	research, development, test, and evaluation
REC	record of environmental consideration
Rep	repetition
RH	relative humidity
RPD	relative percent difference
RSD	relative standard deviation
RTD	resistance temperature detector
RTM	real-time monitor
SAR	safety assessment report
SD	standard deviation
SDS	safety data sheet
SEP	system evaluation plan
sLpm	standard liters per minute
sm^3	standard cubic meters
SME	subject-matter expert
SOP	standing operating procedure
SPS	system performance specification
SSP	system support package
SSPL	SSP list
SWIFT	swatch including filter test
T&E	test and evaluation
TB	technical bulletin
TCD	thermal conductivity detector
TECMIPT	Test and Evaluation Capabilities and Methodologies Integrated Process Team
TECN	test and evaluation capabilities and needs
TEP	triethyl phosphate
TI	technical instruction
TIC	toxic industrial chemical
TICN	test item control number
TM	technical manual
TMP	trimethyl phosphate

APPENDIX E. ABBREVIATIONS.

TO	technical order
TOP	Test Operations Procedure
TPP	tripropyl phosphate
TRR	test readiness review
TWA	time-weighted average
UCL	upper control limit
USACHPPM	U.S. Army Center for Health Promotion and Preventive Medicine
USL	upper specification limit
V&V	verification and validation
VDLS	VISION Digital Library System
VISION	Versatile Information Systems Integrated ON-line
v/v	volume/volume
VX	persistent nerve agent
WVC	water vapor content
w/w	weight/weight

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** The inclusion of SOPs is only to serve as an example of these type procedures that are used at DPG and as a reference for other installations. Many SOPs are specific to a particular installation, facility, or instrument, and may not be applicable between different installations, facilities, or instruments without modifications. It is expected that installations will have their own equivalent SOPs. These equivalent SOPs must be provided to the Test & Evaluation (T&E)

community interested in this test method to properly understand the data produced, any differences between test method application between installations, and therefore, the ability to compare data produced by different installations. If an installation does not have an equivalent SOP already in place, these or other similar procedures could be used as temporary guides until appropriate SOPs are developed. The most current version of these SOPs can be requested through U.S. Army Dugway Proving Ground (DPG) or Edgewood Chemical and Biological Center (ECBC).

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APPENDIX G. APPROVAL AUTHORITY.

CAPABILITY AREA PROCESS ACTION TEAM (CAPAT) ENDORSEMENT

DEPARTMENT OF THE ARMY
OFFICE OF THE DEPUTY UNDER SECRETARY OF THE ARMY
102 ARMY PENTAGON
WASHINGTON, DC 20310-0102

MEMORANDUM FOR

Chemical, Biological, Radiological and Nuclear Defense Test and Evaluation Executive, Office of the Deputy Under Secretary of the Army, Taylor Building, Suite 8070, 2530 Crystal Drive, Arlington, VA 22202

SUBJECT: Test and Evaluation Capabilities and Methodologies Integrated Product Team (TECMIPT) Recommendation for Test Operations Procedure (TTOP) 08-2-197 Chemical Protection Testing of Sorbent-Based Air Purification Components (APCs)

1. The Collective Protection Capability Area Process Action Team (CAPAT) has completed its review of the subject TTOP in accordance with the Chemical and Biological Program Test and Evaluation Process, 18 April 2014. All signatory members of the CAPAT have provided their concurrences to the TTOP (enclosed).

2. Based on the concurrence of the CAPAT, I recommend the CBRN Defense T&E Executive endorse this TTOP as a Department of Defense Test and Evaluation Standard.

OBRIEN.SEAN.P.1230553501

Encl

SEAN P. O'BRIEN
TECMIPT Chair

APPENDIX G. APPROVAL AUTHORITY.

CAPABILITY AREA PROCESS ACTION TEAM (CAPAT) ENDORSEMENT

TECMIPT Test Operations Procedure (TTOP) 08-2-197 Chemical Protection Testing of Sorbent-Based Air Purification Components (APCs)

Collective Protection Capability Area Process Action Team (CAPAT):

Darren Jolley, U.S. Army Dugway Proving Ground (DPG)
Mark Hanning-Lee, DPG
Sun McMasters, DPG
Amy Maxwell, U.S. Army Edgewood Chemical Biological Center (ECBC)

CAPAT Review & Concurrence: February 2015

Test and Evaluation Capabilities and Methodologies Integrated Process Team (TECMIPT) Participants:

DISTRIBUTION A. Approved for public release: distribution unlimited.

REFERENCES:
(a) *Chemical and Biological Defense Program (CBDP) Test and Evaluation (T&E) Standards Development Plan*, dated 19 July 2010.
(b) *Memorandum of Understanding (MOU) Among the Department of National Defence of Canada the Secretary of State for Defense of the United Kingdom of Great Britain and Northern Ireland and the Secretary of Defense on Behalf of the Department of Defense of the United State of America concerning the Research, Development and Acquisition of Chemical, Biological and Radiological Defense Materiel*, dated June 2000. Amendment One, dated August 2006.

APPENDIX E. ABBREVIATIONS.

CBRN DEFENSE T&E EXECUTIVE ENDORSEMENT

DEPARTMENT OF THE ARMY
OFFICE OF THE DEPUTY UNDER SECRETARY OF THE ARMY
102 ARMY PENTAGON
WASHINGTON, DC 20310-0102

DUSA-TE

6 Nov 2015

MEMORANDUM FOR DISTRIBUTION

SUBJECT: TECMIPT Test Operations Procedure (TTOP) 08-2-197 Chemical Protection Testing of Sorbent-Based Air Purification Components (APCs)

1. Reference: Memorandum, DUSA-TE, and 19 July 10, subject: Chemical and Biological Defense Program (CBDP) Test and Evaluation (T&E) Standards Development Plan.

2. TTOP 08-2-197 was developed, coordinated, and approved by the members of the Collective Protection Capability Area Process Action Team (CAPAT) in accordance with the reference. The U.S. Army Test and Evaluation Command (ATEC) approved according to their TOP approval process.

3. I endorse this TTOP as a DoD T&E Standard for decontamination testing and encourage its broad use across all test phases. All T&E Standards are for government associated program access and use. They are stored in Army Knowledge Online (AKO), located at (https://www.us.army.mil/suite/files/22142943), on the National Institute of Standards and Technology (NIST) website (http://gsi.nist.gov/global/index.cfm/L1-4/L2-19/A-664), and the TECMIPT (http://www.amsaa.army.mil/TECMIPT/Standards.html).

4. My point of contact for this action is Ms. Deborah Shuping, (703) 545-1119, deborah.f.shuping.civ@mail.mil.

Encl

PATRICK L. WALDEN
Colonel, IN
Acting CBRN Defense T&E Executive

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APPENDIX G. APPROVAL AUTHORITY.

CSTE-TM 24 June 2016

MEMORANDUM FOR

Commanders, All Test Centers
Technical Directors, All Test Centers
Directors, U.S. Army Evaluation Center
Commander, U.S. Army Operational Test Command

SUBJECT: Test Operations Procedure (TOP) 08-2-197 Chemical Protection Testing of Sorbent-Based Air Purification Components (APCs), Approved for Publication

1. TOP 08-2-197 Chemical Protection Testing of Sorbent-Based Air Purification Components (APCs), has been reviewed by the U.S. Army Test and Evaluation Command (ATEC) Test Centers, the U.S. Army Operational Test Command, and the U.S. Army Evaluation Center. All comments received during the formal coordination period have been adjudicated by the preparing agency. The scope of the document is as follows:

This TOP provides preparation, planning, conducting, and reporting procedures for testing APCs for the protection capability area of chemical defense. These procedures are designed to analyze the effectiveness of APCs for removing any chemical gas or vapor from incoming air. he intent of this process is to produce traceable, quantifiable, and defensible data that can be used to analyze an APC's ability to filter air in a chemically contaminated environment.

2. This document is approved for publication and will be posted to the Reference Library of the ATEC Vision Digital Library System (VDLS). The VDLS website can be accessed at https://vdls.atc.army.mil/.

3. Comments, suggestions, or questions on this document should be addressed to U.S. Army Test and Evaluation Command (CSTE-TM), 2202 Aberdeen Boulevard-Third Floor, Aberdeen Proving Ground, MD 21005-5001; or e-mailed to usarmy.apg.atec.mbx.atec-standards@mail.mil.

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RAYMOND G. FONTAINE
Director, Test Management Directorate (G9)

Forward comments, recommended changes, or any pertinent data, which may be of use in improving this publication to the Range Infrastructure Division (CSTE-TM), U.S. Army Test and Evaluation Command, 2202 Aberdeen Boulevard, Aberdeen Proving Ground, Maryland 21005-5055. Technical information may be obtained from the preparing activity: Commander, West Desert Test Center, U.S. Army Dugway Proving Ground, ATTN: TEDT-DPW, Dugway, UT 84022-5000. Additional copies can be requested through the following website: : http://www.atec.army.mil/publications/topsindex.aspx, or through the Defense Technical Information Center, 8725 John J. Kingman Rd., STE 0944, Fort Belvoir, VA 22060-6218. This document is identified by the accession number (AD No.) printed on the first page.